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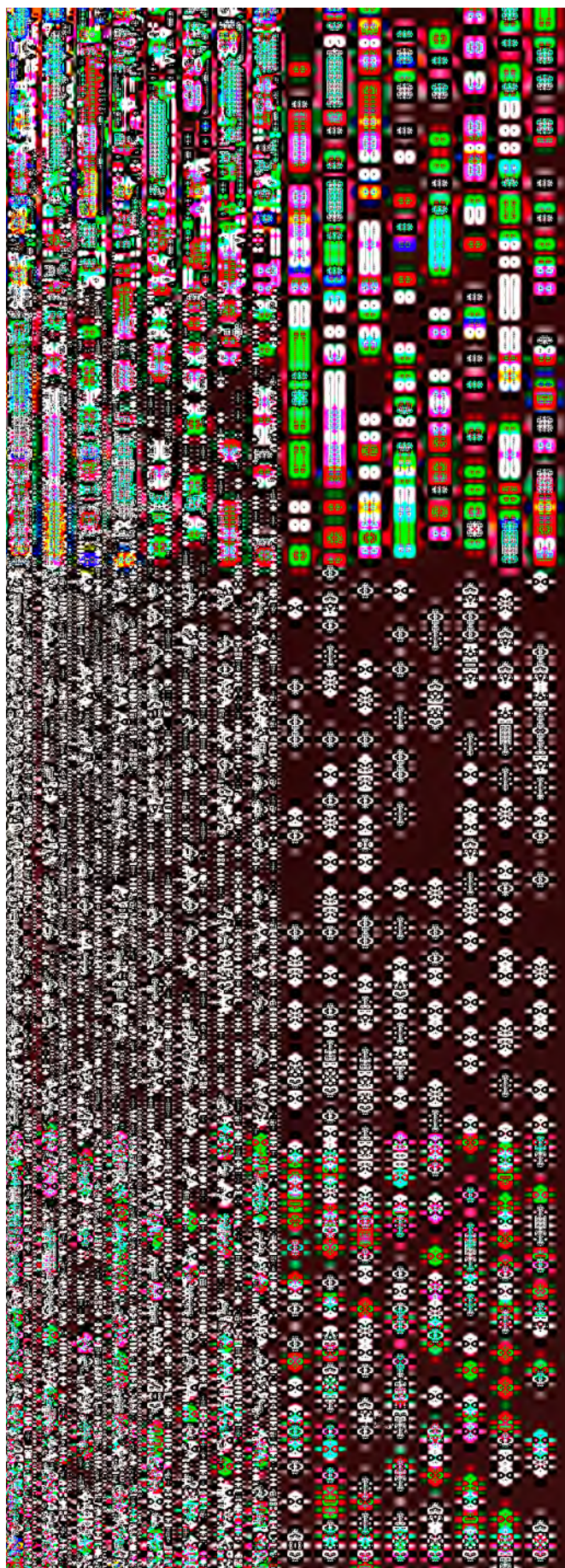
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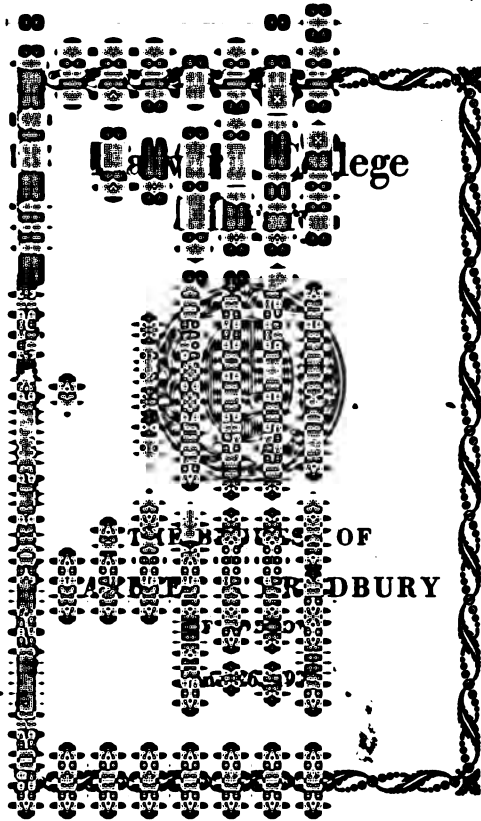
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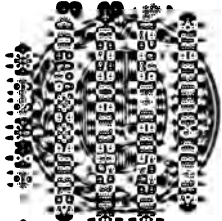
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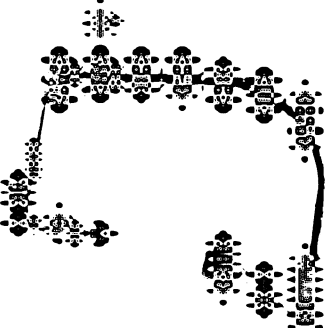


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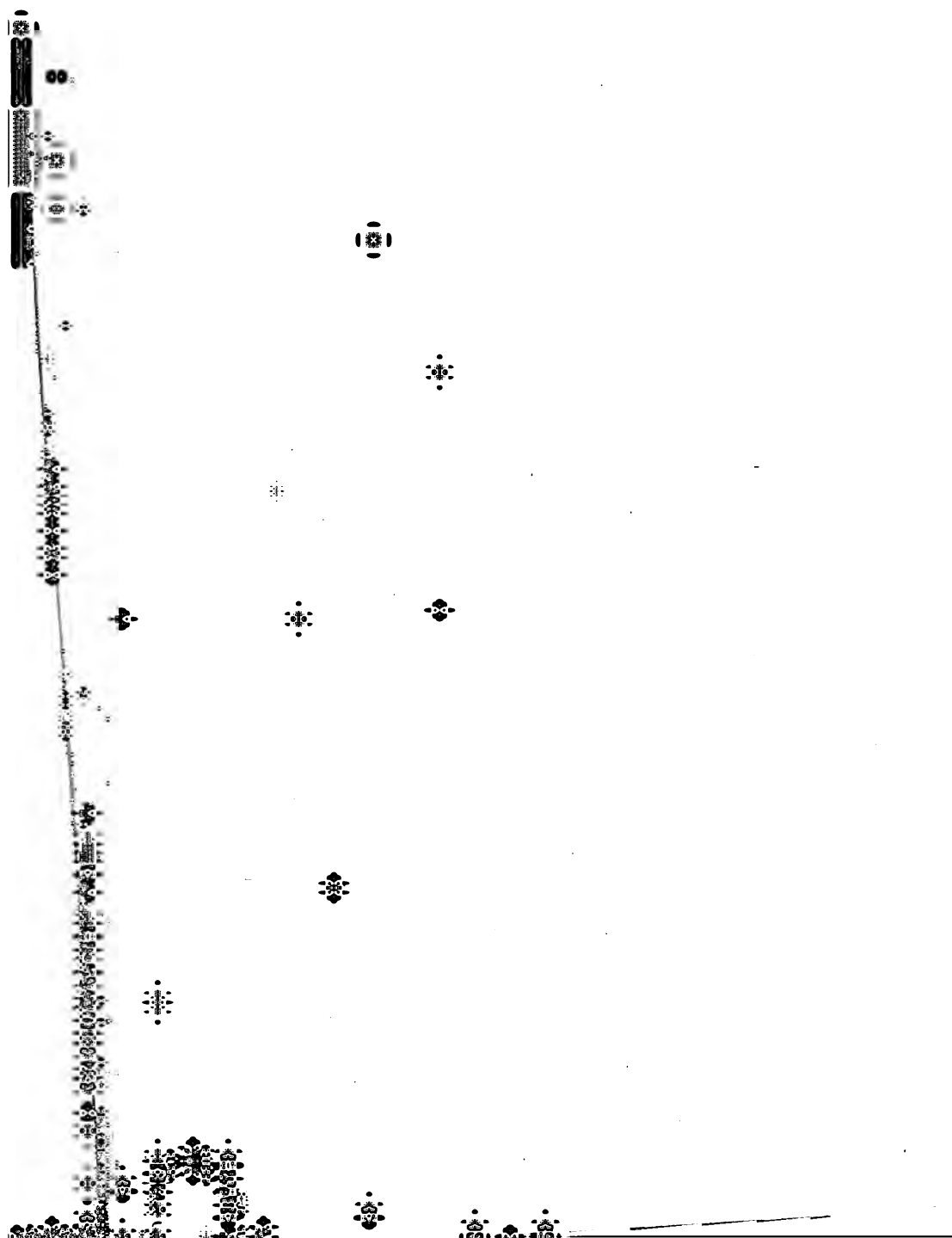
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analysis of unknown solutions

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OF CHEMISTRY.

It is not necessary for the class to subject.

space, as wood, water and air, which is called its *extension*, properties, including *gravitation*, and *divisibility*, which are things which exist but are not, mental processes and the

er, like iron, marble, water or the general properties of matter to distinguish it from other *properties*. Among these are for a gas, its density, and some melting-point, boiling-point, tenacity, behavior toward properties. The particular compounds by a substance serve to separated into several other, and a substance is said to are referred to, collectively,

ually undergoing change. The nothing is permanent. The may all be classified into two condition of the matter and the

The changes which affect changes and include all forms of changes, changes of state, many others. In a physical undergoing the change is not the composition of matter are all burnings or combustions, matter, the rusting of iron, and

various others. A chemical change is frequently called a *chemical reaction* or simply a *reaction*. In a chemical change the identities of the matter undergoing the change are lost and new substances with new identities are formed.

Physical and chemical changes are often mutually dependent upon each other; heat, light, electricity, solution and other physical agencies, often produce chemical changes and, conversely, chemical changes often produce heat, light, electricity and other physical effects. Physical and chemical changes are all caused by *energy*, which is that which can produce change in matter. Energy cannot be created or destroyed, which fact is referred to as the *conservation of energy*. Heat, light, electricity, and the power due to the motion or the position of matter, are different forms of energy, and these agencies may produce chemical change or be produced by chemical change. Matter can neither be created nor destroyed, which fact is referred to as *indestructibility* or the *conservation of matter*.

Physical science studies the properties of matter and the effects of energy on matter. *Physics* is the branch of physical science which studies the general properties of matter and the changes which matter undergoes without affecting its composition. *Chemistry* is the branch of physical science which studies the specific properties and composition of substances and changes in their composition.

Laws of Definite and Multiple Proportions.—Early in the development of the science of chemistry two important laws were formulated from the results of experiments. The first is called the *law of definite proportions* and may be stated as follows: In any chemical change there is always a definite relationship between the amounts of the substances entering into and produced by the change. For example, copper and sulphur, when heated together, will react upon each other, in the proportion of 63 parts of copper to 32 parts of sulphur, any excess of either substance being left unchanged. Also, there is a definite amount of the resulting substance formed by the reaction of any given amount of copper upon the corresponding amount of sulphur.

The second law is called the *law of multiple proportions* and may be stated as follows: If two substances react upon each other in different proportions by weight the amounts of each substance in the different reactions bear a simple relationship to each other. For example, sulphur will react upon oxygen in two ways: in one of the reactions a given amount of sulphur, as 32 parts, reacts upon 32 parts of oxygen, and in the other 32 parts of sulphur reacts upon 48 parts of oxygen. In these two different reactions, the amounts of the sulphur being equal, the relative amounts of the oxygen are to each other as two to three, a simple relationship.

is a separate portion of the whole, the water in a reservoir, the mass may be divided into two parts. This division were carried far enough, the water could be obtained which could not change the identity of the molecules. They are so small that even in a small mass of water no machinery fine enough to separate the molecules, nor is it possible to change them. The specific properties of its molecules and their

united by other than mechanical forces, the identity of the particles which are contained in the indivisible particles of matter is united with each other to last not only for an instant as in the case of mechanical union. Any collection of molecules united by other than mechanical forces for each other is called a chemical compound, which causes atoms to unite by the force of *chemical affinity*, *chemical*

of matter is a brief statement which is the foundation of many applications in physics. It is proved, it is the only commodity that has been formulated, and it is true.

1. If the particles of a substance are all
 2. the same, the substance is of
 3. one kind and cannot be separated into any
 4. other. Such a simple substance
 5. has not yet been discovered,
 6. all the elements known being composed

are all exactly alike, but each is, the substance is called a compound. The number of words formed by the process appears to be unlimited. And many others are manufactured by the constituents of a process, but a compound

may be separated into simpler compounds or elements by chemical processes, such a separation being called a *chemical decomposition*. In a compound the constituents which compose it are united with each other in a fixed and definite proportion by weight. The compound has a separate identity, and properties which are very different from those of its constituents.

If a substance is formed of different kinds of molecules it is called a *mixture*. In a mixture the different constituents are merely commingled with each other, and each retains its own identity and properties, and the properties of the mixture are between the properties of the constituents. It is generally possible to separate the constituents of a mixture from each other by mechanical processes, because of their different solubilities, melting-points or other specific properties, and the relative amounts of the constituents may vary.

Many natural substances, like granite and air, are mixtures, while others, like water, salt and marble, are compounds, and a few, like gold and sulphur, are elements. An absolutely pure compound or element is very unusual, the impurities being mixed with the compound or element.

An element is said to be in the *nascent state* for the instant during which it is being set free from a compound and before its atoms have united with each other to form molecules. When the atoms of an element have united to form molecules, a certain amount of energy is necessary to overcome their attraction for each other, so an element is more active, chemically, in the nascent state, as its atoms then exhibit their full chemical affinities.

Kinds of Chemical Changes.—*Combination*, when two or more substances unite with each other chemically to produce one substance, as the burning of hydrogen in oxygen to form water.

Decomposition, when one substance forms two or more substances as the result of a chemical change, as the decomposition of water by an electric current to form hydrogen and oxygen.

Double decomposition, when two substances react upon each other to produce two other substances by an exchange of constituents. This is the commonest kind of chemical change, and many examples of it will be studied.

There are many changes which belong to more than one of these kinds of chemical changes, one, called *substitution*, being quite common. In this kind of a change an element reacts upon a compound in such a way that a part of the compound is displaced, the first element taking its place, as the reaction between zinc and sulphuric acid, in which the zinc replaces the hydrogen of the acid, forming zinc sulphate and setting hydrogen free.

Symbols.—Early in the development of the science of chemistry it was found convenient to designate elements by signs. At first these were astronomical signs and geometrical figures, but as the number of known elements increased this method was found to be faulty and the present *symbols* came into use. The symbol of an element is formed from the English or some other name of the element by taking the first letter, alone, or with some other letter in the name. The first letter of a symbol is always a capital, and if there is a second letter, it is always a small letter. When symbols first came into use they were mere abbreviations of the names of the elements, and they are sometimes used in this way at present. In most cases, however, they stand for one atom of an element. If several atoms are to be indicated the symbol is multiplied by methods discussed in the next paragraph.

Formulas.—Chemical *formulas* are used to represent molecules. Formulas are made by writing the symbols of the elements in the molecules, one after the other. If a molecule contains only one atom of a certain element the symbol of that element indicates this; more than one atom of any element in a molecule is indicated by an inferior figure written after the symbol of that element, thus H_2O is the formula for water and tells us that each molecule of water contains two atoms of hydrogen and one atom of oxygen; H_2SO_4 is the formula for sulphuric acid, whose molecules each contain two atoms of hydrogen, one atom of sulphur and four atoms of oxygen. When for any reason a part of a molecule consisting of more than one atom is considered as a group to be multiplied, it is enclosed in a parenthesis, which is followed by the inferior figure, as in ammonium sulphate $(\text{NH}_4)_2\text{SO}_4$, whose molecule contains two NH_4 groups, combined with the SO_4 group. A formula generally represents one molecule of a compound. More than one molecule are shown by a line figure written before the formula, thus two molecules of ammonium sulphate are written $2(\text{NH}_4)_2\text{SO}_4$.

Chemical Equations.—When a chemical change occurs, it is frequently possible to express what occurs by the use of symbols and formulas written in the form of an equation. Besides being of great convenience, these equations express exact relationships between the quantities of the substances entering into the reaction and produced by it; thus the decomposition of water is expressed $2\text{H}_2\text{O} = 2\text{H}_2 + \text{O}_2$, which tells us that when water is decomposed two molecules yield two molecules of hydrogen and one molecule of oxygen. Actually very many molecules are involved when any appreciable amount of water is decomposed. This equation is sometimes written $\text{H}_2\text{O} = 2\text{H} + \text{O}$, but the first form is preferable, as it is the simplest way of writing all atoms combined to form molecules, which they do as soon as they are liberated from other molecules.

Chemical equations, like mathematical equations, must be balanced, that is, they must have the same number of atoms, of each kind involved, on both sides of the equality sign.

Atomic Weight.—Atoms are so extremely small that their absolute weights, in terms of any ordinary unit, are very small decimals which have been calculated, approximately, but they cannot be determined directly. The relative weights of the atoms of different elements have been quite accurately determined and have been referred to the weight of an atom of hydrogen as a unit. What is known as the *atomic* or *combining weight* of an element is the number of times that the weight of its atom is heavier than the weight of an atom of hydrogen. Thus the atomic weight of oxygen is 15.88, which means that an atom of oxygen is 15.88 times as heavy as an atom of hydrogen; the atomic weight of iron is 55.5, which means that an atom of iron weighs 55.5 times as much as an atom of hydrogen, and so on. The sum of the weights of the atoms in a molecule of a compound is the *molecular weight* of that compound.

Valence.—The atoms of different elements differ from each other in the number of atoms of other elements that they can combine with chemically to form molecules. This quantity of combining power of the atoms of an element is called its *valence* and is measured by the number of atoms of hydrogen that one atom of the element can combine with, or take the place of. The unit of valence is called a *bond*. An element whose atom combines with, or displaces, one atom of hydrogen, as Cl in HCl and Na in NaCl, has one bond and is called a monad element or simply a *monad*. An element whose atom combines with, or displaces, two atoms of hydrogen, as O in H₂O and Ca in CaO, has two bonds and is called a *dyad*. Similarly a *triad* has three bonds; a *tetrad*, four; a *pentad*, five; a *hexad*, six. There are very few compounds in which an element appears to have a valence greater than six. The valence of an element is indicated, when necessary, by the use of superior Roman numerals written after the symbol of the element, as H', O'', C^{iv}, etc.

Some of the elements have the same valence in all of their compounds, while others vary in valence, forming two or more series of compounds. When atoms combine to form molecules all bonds must be satisfied, the number of atoms in each molecule being the smallest number that will comply with this general law. If two elements have the same valence, one atom of each will form a molecule of their compound, as two monads in H'Cl', Na'Cl', two dyads in Fe''O'', Ca''S'', etc. Two atoms of a monad combine with one atom of a dyad, as in H'₂O''; three atoms of a monad with one atom of a triad, as in N'''H'₃; two atoms of a dyad with one atom of a tetrad, as in C^{iv}O''₂; two atoms of a triad with three atoms

a pentad with five atoms of

ounds containing hydrogen and are called *acids*. They certain dyes, like litmus, contain oxygen combined with the hydrogen and oxygen is called the *characteristic* compounds, called bases, combined with another of the base. An acid will combine with an element of a base forming a *salt*. This reaction is called *neutralization*. Examples: $\text{Ca} + 2\text{HCl} = \text{CaCl}_2 + 2\text{H}_2\text{O}$. In other ways.

When an element combines with oxygen and hydrogen, it is called electro-positive. An element which combines with oxygen and hydrogen, to form an acid, is called electro-negative. The classification of elements has long been used and was first proposed by Lavoisier. It is not exact, as there are many elements which are metals and non-metals under different conditions. In degree, some elements are more metallic than others, and calcium, some always are, and a few forming both. There are about fifty metals, and many of them are familiar substances, like iron, copper, and silver, seldom seen in the free state, but they are easily obtained from their compounds. Some elements, like sodium, are so active when they are freshly cut that they burn with both heat and electricity. Some elements, like zinc, iron, silver, copper, and gold, are important metals. An alloy is a mixture of two or more metals together. Some alloys are harder than the metals themselves, and others appear to be mixtures. Some alloys, such as brass,

are harder than the metals do. Some elements, like oxygen, sulphur, chlorine, carbon, silicon and boron, are non-metals. Replaceable hydrogen in the acids is called the *acidic* hydrogen, and acids with two, three and four replaceable hydrogens are called *di-basic*, *tri-basic*, and *tetra-basic*. There are few acids with

more than four replaceable hydrogen atoms in the molecules. An acid composed of hydrogen and one other element and no oxygen is called a *hydracid*. These are few in number: hydrochloric acid, HCl; hydrobromic acid, HBr; hydriodic acid, HI; hydrofluoric acid, HF; and hydrosulphuric acid, H_2S , comprising the entire list of hydracids. Nearly all of the many other acids contain oxygen in addition to the hydrogen and the characteristic element and are called *oxyacids*. An *acid anhydrid* is a compound of a negative element with oxygen which will combine with water to form an acid, or which is formed, along with water, when an acid is decomposed, as SO_3 combines with water to form H_2SO_4 ; and CO_2 is formed, along with H_2O , on the decomposition of H_2CO_3 . Different acids may be obtained from the same negative element, if it combines with different amounts of oxygen to form different anhydrides, or one of its anhydrides combines with different amounts of water.

A base may be either a compound of a metal and oxygen or of a metal, oxygen and hydrogen. A base that is soluble in water is called an *alkali*. Alkalies turn red litmus back to blue. Dyes, like litmus, whose colors are affected by acids and alkalies, are called *indicators*. A substance which affects an indicator like an acid is said to have an *acid reaction*. A substance which has the opposite effect on an indicator, behaving like an alkali, is said to have an *alkaline reaction*. Substances which do not affect the color of indicators are said to be *neutral* in reaction.

Salts formed by the complete neutralization of an acid by a base, in which all of the hydrogen of the acid is replaced by a metal, are called *normal salts*, as Na_2SO_4 from H_2SO_4 . Such salts are generally neutral in reaction. A salt in which the base has not entirely neutralized an acid, and the metal has replaced only a part of the hydrogen of the acid, is called an *acid salt*, as NaHSO_4 . They are generally acid in reaction. A salt in which the acid has not entirely neutralized the base, and which retains a part of the oxygen, or oxygen and hydrogen of the base, is called a *basic salt*, as BiOCl . A *double salt* is a salt that contains more than one metal, as KNaSO_4 .

Compound Radicals.—There are certain groups of atoms, called *compound radicals*, which behave like single atoms. They do not exist separately, but one of them may be found in several different compounds, and will pass from one compound to another without its parts becoming separated. A compound radical has a definite valence, which is the difference between the valences of its constituent atoms. Some are electro-positive and behave like metals, and some are electro-negative and behave like non-metals. Many of these groups have commonly used names, like hydroxyl (OH)', ammonium (NH_4)', and cyanogen (CN)'. From acids there is an important class of such groups, called *acid radicals*, and found in the

salts of the acids, each consisting of all of an acid excepting the replaceable hydrogen. For example, $(\text{NO}_3)'$, from HNO_3 , is found in salts of nitric acid; $(\text{SO}_4)''$, from H_2SO_4 , in salts of sulphuric acid; $(\text{PO}_4)'''$, from H_3PO_4 , in salts of phosphoric acid, and so on.

Nomenclature.—Systematic rules have been gradually adopted for naming chemical compounds, and a knowledge of them is important, as it enables one to name a compound from its formula, or to give the formula from its name. These names are confusing, as many compounds, like water, salt and marble, have long been known as separate substances and bear common names, called *synonyms*, which have no relationship to their chemical composition; also, the rules have been changed and improved from time to time, but compounds are often called by names given to them under old rules.

The only rule governing the naming of elements is that “-um” indicates a metal, though there are several important metals, like iron, copper and gold, whose commonly used names do not conform to this rule. Some of the elements, like gold, sulphur and iron, have been known for a long time and their names have long been used. Those discovered more recently have been named from some striking property, or from the localities in which they were first found.

Many of the metals vary in valence and form two distinct series of compounds. These are distinguished from each other by changing the end of the name of the metal to *-ous* for the compounds in which the metal has the lower valence and to *-ic* for the compounds in which it has the higher valence. Examples: compounds of mercury in which the mercury has a valence of one are the mercurous compounds, and compounds in which mercury has a valence of two are the mercuric compounds; compounds of iron (ferrum) in which the iron has a valence of two are the ferrous compounds, and compounds in which iron has a valence of three are the ferric compounds.

A *binary compound* is a compound containing two elements only, like HCl , H_2O , etc. A binary compound is called by the name of the positive element in the compound followed by the name of the negative element with its last part changed to *-ide*. Example: NaCl is sodium chloride. If two elements form two or more compounds with each other, these compounds may sometimes be distinguished by the rule for distinguishing different series of compounds of metals. Examples: HgCl is mercurous chloride and HgCl_2 is mercuric chloride. Or prefixes are used to indicate the number of atoms of the negative element in a molecule of the compound, the number of atoms of the positive element remaining the same. Examples: H_2O is hydrogen monoxide; H_2O_2 is hydrogen dioxide. Compound radicals are often considered as elements, and their compounds may be named like binary compounds. Examples: NH_4Cl is ammonium chloride; $\text{Hg}(\text{CN})_2$ is mercuric

cyanide. Bases are named by the binary rule, thus CaO is calcium oxide and $\text{Ca}(\text{OH})_2$ is calcium hydroxide.

Hydracids are named by taking the name of the characteristic element, changing its last part to *-ic* and prefixing *hydro*. Examples: HCl is hydrochloric acid; H_2S is hydrosulphuric acid. By the binary rule these would be hydrogen chloride and hydrogen sulphide, respectively, and such names are sometimes used.

Each negative element generally forms several oxyacids. In naming the oxyacids formed by an element, one containing a relatively large number of oxygen atoms in its molecules is called by the name of the element with its last part changed to *-ic*. Example: HClO_3 is chloric acid. The acid containing the next smaller number of oxygen atoms in its molecules is named by taking the name of the characteristic element and changing its last part to *-ous*. Example: HClO_2 is chlorous acid. If there is an acid with still less oxygen than in the "ous" acid it is called by the name of the characteristic element, changing its last part to *-ous* and prefixing *hypo*. Example: HClO is hypochlorous acid. If there is an acid with more oxygen in the molecules than is contained in the "ic" acid, it is called by the name of the characteristic element, changing its last part to *-ic* and prefixing *per*-. Example: HClO_4 is perchloric acid.

Salts of hydro-acids are binary compounds and are named by the binary rule. Example: CaCl_2 is the calcium salt of hydrochloric acid and is named calcium chloride. Salts of oxyacids are named from the names of the acids. A salt of an acid whose name ends in *-ic* is called by the name of the positive element or radical followed by the name of the acid with its last part changed to *-ate*. Examples: KClO_3 is the potassium salt of chloric acid and its name is potassium chlorate; similarly KClO_4 is potassium perchlorate. A salt of an acid whose name ends in *-ous* is called by the name of the positive element, followed by the name of the acid with its last part changed to *-ite*. Examples: KClO_2 is the potassium salt of chlorous acid and its name is potassium chlorite; similarly KClO is potassium hypochlorite. Salts were formerly named in reverse order and the name of the metal modified in ways no longer in good usage, but these older names are often seen. Examples: sodium chloride was formerly called chloride of soda; potassium chlorate was formerly called chlorate of potash, and so on.

Acid salts are generally designated by the prefix *bi*-, as in sodium bi-carbonate, which is NaHCO_3 , sometimes called sodium hydrogen carbonate or sodium acid carbonate. Basic salts are commonly designated by the prefixes *sub*- or *oxy*-, as in bismuth subchloride, which is BiOCl , sometimes called bismuth oxychloride or bismuth basic chloride. The name of a double salt contains the names of both metals in the salt, as in potassium-sodium sulphate, KNaSO_4 .

es are not included in the separately as the compounds

nt into contact with liquids, or less extent. A solid substance may lose its solid condition, when it is said to be *dis-* at part of the solid may be form of small solid particles. to be *soluble* in the liquid, will not dissolve in a liquid ough this is a comparative in liquids to some extent. ry slightly soluble. When that it can dissolve at the id to be *saturated*. If the ve more of a solid than at mount is thus dissolved the excess being deposited from ases dissolve in liquids to a that solids do. When two ortions they are said to be hich is of great importance ing operations.

sediment; when an insoluble ical reaction or other means is called *precipitation*. If to form an insoluble sub- do so. This is called the e of it in chemical processes. from a liquid we generally k. This consists of pouring n a smooth folded paper clear liquid which passes he folded paper should not and it is generally better to if a watery liquid is to be ar it will often do so if it is lter containing the precipi-

wn, but it has been carefully ht it appears, merely, that influences of the molecules tion between the molecules molecules of a gas, disappear.

Some solutions appear to be of this character, but in others there is a much more radical change.

If two fluids, liquids or gases, which are miscible, are brought into contact with each other, they immediately begin to mix, and this spontaneous mixing will continue until a uniform mixture results. It is rapid for gases and slow for liquids. When the liquids are only superimposed, one on the other, the process is called *diffusion*; when they are separated by a porous membrane the process is called *osmosis*. Osmosis is a selective process when applied to solutions, some substances in solution passing through the porous membrane with the solvent, while others do not pass through. The substances which will osmose with their solvents are called *crystalloids*, and those that will not osmose are called *colloids*. *Dialysis* is the process of separating crystalloids from colloids by osmosis. Colloids and crystalloids are in quite different conditions in their solutions. Gelatin, starch, gums, soap and rubber are colloids with most solvents, also certain hydroxides and other compounds of metals, such as are found in milk of magnesia and dialyzed iron. Colloids do not form true solutions, as the molecular influences are not overcome. They appear to swell and absorb the solvent to form a pasty or liquid mixture. The boiling- and freezing-points of the solvent are not changed, as they are in true solutions.

When crystalloids dissolve there may be only a molecular dispersion, as in the solution of sugar in water, but this is exceptional. In nearly all solutions of crystalloids the molecules of the dissolved substances are not only dispersed, but they are partially broken down, or *dissociated*, into positive and negative parts called *ions*. Example: in the solution of sodium nitrate some of the molecules of NaNO_3 are separated into the electro-positive ion Na and the electro-negative ion NO_3 . The relative number of molecules thus disassociated varies with the identity of the compound, the solvent, the temperature and the strength of the solution. In weak solutions the dissociation is relatively greater than in strong solutions. The electrical conductivity of solutions is due to the positive and negative characters of the ions. When a compound is decomposed by an electric current the process is called *electrolysis*.

Water of Crystallization.—Many chemical compounds tend to assume characteristic geometric forms, called *crystals*, when the compounds are deposited from a solution as the solvent evaporates, or when the compound solidifies after fusion, or is condensed from the gaseous condition. A *crystalline compound* is one which is in the form of crystals, and an *amorphous compound* is one which is not in the form of crystals. The crystals of a compound are generally alike in form, though they may vary widely in size, and they may retain some loosely combined water, which is called *water of crystal-*

will not form crystals or the crystalline form has a $\text{O}_5\text{H}_2\text{O}$. If this is heated the residue of CuSO_4 is a substance, however, contain water of crystallization, sodium chloride, readily without any water in the

in the molecules of a substance. If the water of crystallization by heating, the process is then said to be *anhydrous*. Its water of crystallization substance which absorbs and if the water appears partially or completely, the crystalline washing soda, $\text{CO}_3\text{10H}_2\text{O}$ and effloresces powder. Anhydrous copper sulphate, changing from white to blue, CaCl_2 , is a deliquescent substance, $\text{CaCl}_2\text{6H}_2\text{O}$, and then absorb water, ultimately dissolving in the

crystallization is dissolved in the solvent, becomes a part of the solvent, and the formula of the compound, if not dissolved the water of crystallization.

Example: alum crystals contain nearly one-half water, and referring to alum, unless the water of crystallization.

A substance into its constituent parts, *analysis* is the identification of the constituents of a substance.

Quantitative analysis is the analysis of the constituents of a substance, to be used to ascertain the amount of each or to find the presence or absence.

A substance has certain *characteristic* properties which identify it. Any substance used in a chemical reaction is a *reagent*. If it is used in a chemical reaction as a *test-solution*. A list of the strengths will be found

THE METALS.

It is customary to separate and identify the metals in a substance before looking for the acid radicals and non-metals. The scheme generally used for the separation and identification of the metals depends upon the fact that a large majority of the metals, in solutions of their compounds, will form precipitates with hydrogen sulphide, H_2S , under various conditions, these precipitates varying widely in their properties. In carrying out this scheme it is necessary that the substances be in solution, and, at the beginning of the study of qualitative analysis, the work is done upon solutions of known and unknown composition. Afterward, some practice on the bringing of substances into solution is necessary.

In this manual the principal metals are classified into seven groups, as follows:

Group 1.—Metals precipitated as chlorides by hydrochloric acid, HCl : silver, Ag ; mercurous mercury, Hg' ; and all but a very small amount of lead, Pb .

Group 2.—Metals precipitated as sulphides by hydrogen sulphide, H_2S , in the presence of hydrochloric acid, HCl , and whose sulphides are insoluble in ammonium sulphide, $(NH_4)_2S_x$: mercuric mercury, Hg'' ; bismuth, Bi ; copper, Cu ; cadmium, Cd ; and the small amount of lead not removed with group 1.

Group 3.—Metals precipitated as sulphides by hydrogen sulphide, H_2S , in the presence of hydrochloric acid, HCl , and whose sulphides are soluble in ammonium sulphide, $(NH_4)_2S_x$: arsenic, As ; antimony, Sb ; tin, Sn ; gold, Au ; and platinum, Pt .

Group 4.—Metals precipitated as hydroxides by ammonium hydroxide, NH_4OH , in the presence of ammonium chloride, NH_4Cl : iron, Fe ; chromium, Cr ; and aluminum, Al .

Group 5.—Metals precipitated as sulphides by ammonium sulphide, $(NH_4)_2S$, or hydrogen sulphide, H_2S , in the presence of ammonium hydroxide, NH_4OH : cobalt, Co ; nickel, Ni ; manganese, Mn ; and zinc, Zn .

Group 6.—Metals precipitated as carbonates by ammonium carbonate, $(NH_4)_2CO_3$, in the presence of ammonium chloride, NH_4Cl , and ammonium hydroxide, NH_4OH : barium, Ba ; strontium, Sr ; and calcium, Ca .

Group 7.—Metals not precipitated by any group reagent: magnesium, Mg ; potassium, K ; sodium, Na ; lithium, Li ; and the compound radical ammonium, $(NH_4)'$.

precipitate the groups are called precipitate the metals of the metals, each of the group order given. If there is no precipitate is added, that group is precipitated. If there is a precipitate precipitation appears to be removed out of the liquid, and the amount of the last used reagent, is complete; and it is then tested

the text-book are to be carried out. Directions are given, from one solution to be tested is poured into test-tubes of the reagent are added, and it is necessary to completely precipitate. Diluted acids and solutions are otherwise specified.

Analysis depends very largely on the reactions obtained. This ability to perform much practice is necessary to obtain known solutions. The analysis of unknown solutions

hydrochloric acid, HCl.

Hg⁺; silver, Ag.

$\text{At} = 207.10$.

state, alone and in many compounds. The compounds of lead are yellow, red or brown. Lead is soluble in water, but all other compounds are only very slightly soluble. Lead is a very poisonous poison, in single large doses, in a short period of time.

TESTS OF LEAD.

"Sugar of lead," $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$.
"white lead," "cerussa,"

PbO_2 .

Lead iodide, PbI_2 .

Lead nitrate, $\text{Pb}(\text{NO}_3)_2$.

Lead oxide, *Plumbi oxidum*, U. S. P., "litharge," PbO .

Red lead oxide, "minium," "red lead," Pb_3O_4 .

Lead dioxide, lead peroxide, PbO_2 .

Lead sulphide, "galena," PbS .

TESTS FOR LEAD.

Use a separate portion of a solution of lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$, or of lead nitrate, $\text{Pb}(\text{NO}_3)_2$, for each of the following tests:

1. Add HCl , obtaining a white precipitate of lead chloride, PbCl_2 .

This precipitate is slightly soluble in water, so the lead is not all precipitated, and a weak solution of a lead salt will not give the reaction. Pour a portion of the liquid containing the precipitate of PbCl_2 on a filter, pass hot water through the filter and the precipitate will dissolve.

Soluble chlorides, as KCl , will give the same precipitate.

2. Add KI , obtaining a yellow precipitate of lead iodide, PbI_2 .

3. Pass H_2S gas through the lead solution, obtaining a black precipitate of lead sulphide, PbS .

$(\text{NH}_4)_2\text{S}$ will give the same precipitate.

4. Add H_2SO_4 , obtaining a white precipitate of lead sulphate, PbSO_4 .

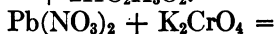
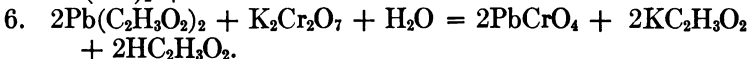
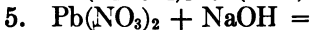
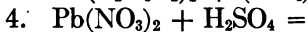
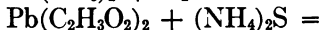
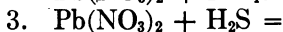
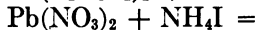
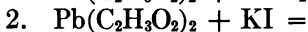
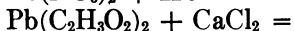
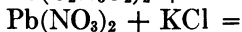
Soluble sulphates, as K_2SO_4 , will give the same precipitate.

5. Add a few drops of NaOH , obtaining a white precipitate of lead hydroxide, $\text{Pb}(\text{OH})_2$, which forms sodium plumbite, Na_2PbO_2 , and dissolves on adding an excess of the NaOH .

6. Add $\text{K}_2\text{Cr}_2\text{O}_7$, obtaining a yellow precipitate of lead chromate PbCrO_4 .

Soluble chromates, as K_2CrO_4 , give the same precipitate.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:



$Hg^i = 200.6.$

QUICKSILVER."

al which is largely used in thermometers and metallurgical some substances, as chalk, that is, divided into minute the metal in this condition y, mercurial ointment and quished metallic mercury. *algams.*

both of which are impor- re, and the mercuric salts compounds vary in color, o black. They are nearly g the only important salt of mercury are poisonous out decomposition.

Mercuric Compounds.

mercuri mite, U. S. P., "calomel," *flavum*, U. S. P., "proto-mercury," HgI .

MERCURY.

mercurous nitrate, $HgNO_3$,
nitrate of mercurous chloride,
ate on a filter and pass hot
ate will not dissolve. Pour
nitrate will blacken, forming
 Hg_2Cl .
ow precipitate of mercurous
obtaining a black precipi-
S, and metallic mercury.
ipitate of mercurous oxide,
lime water, $Ca(OH)_2$. A

mixture formed from calomel and lime water is an old-fashioned remedy, called "black wash."

5. Pour 5 mls of the mercury solution into a small beaker. Place a small piece of copper foil in the solution. The copper becomes covered with a gray deposit of metallic mercury, which brightens when rubbed. Dry the coated copper foil, put it in a dry test-tube and heat gently over the flame of a Bunsen burner. The mercury will volatilize and condense on the cooler part of the test-tube as a gray deposit, which consists of small globules of the metal.

Mercury will deposit from its compounds and amalgamate with many metals as with the copper in this test and care must be taken not to allow mercury or its compounds to come in contact with gold rings.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{HgNO}_3 + \text{HCl} =$
 $2\text{HgCl} + 2\text{NH}_4\text{OH} = \text{NH}_2\text{Hg}_2\text{Cl} + \text{NH}_4\text{Cl} + 2\text{H}_2\text{O}$
2. $\text{HgNO}_3 + \text{KI} =$
 $\text{HgNO}_3 + \text{NH}_4\text{I} =$
3. $\text{HgNO}_3 + \text{H}_2\text{S} =$
 $\text{HgNO}_3 + (\text{NH}_4)_2\text{S} =$
4. $\text{HgNO}_3 + \text{NaOH} =$
 $\text{HgCl} + \text{Ca}(\text{OH})_2 =$
5. $\text{HgNO}_3 + \text{Cu} =$

SILVER (ARGENTUM), $\text{Ag}^1 = 107.12$.

Silver is largely used in the metallic state, being generally alloyed with a small amount of copper to harden it. Its compounds are poisonous and unstable, being easily reduced to the oxide or metallic silver by reducing agents, including organic matter. This reduction is rapid in *actinic* light. Most of the important silver salts are colorless, but the iodide is yellow and the oxide is black. The nitrate is the only important salt which is readily soluble in water.

IMPORTANT COMPOUNDS OF SILVER.

Silver chloride, AgCl .

Silver cyanide, AgCN .

Silver iodide, AgI .

Silver nitrate, *Argenti ntras*, U. S. P., "lunar caustic," AgNO_3 .

Silver oxide, *Argenti oxidum*, U. S. P., Ag_2O .

Silver sulphate, Ag_2SO_4 .

TESTS FOR SILVER.

Use a separate portion of a solution of silver nitrate, AgNO_3 , for each of the following tests:

precipitate of silver chloride,
in ammonia water, NH_4OH ,
AgCl.

precipitate of AgCl on a filter.
the precipitate will not
filter and the precipitate
silver solution and the

precipitate silver as silver chloride.
precipitate of silver iodide,
reagent as potassium silver

precipitate of silver cyanide,
reagent as potassium silver

solution, obtaining a black

precipitate of silver oxide,
ent, but readily soluble in
her.

LOWING EQUATIONS:



ER 1.

p 1 of the metals.

ad, mercurous mercury, or
three of them.

in the solution. If the color
is neutral or acid in reaction,

as it should be. If the color changes to blue add HNO_3 T.S. slowly, with stirring, until the liquid will change the color of blue litmus paper to red.

A. To about 10 mls of the solution contained in a test-tube, add a few drops of HCl T.S. If no precipitate forms, silver and mercurous mercury are absent, and lead, if present, is in very small amount, which will be detected in group 2. If a white precipitate forms it consists of chlorides of metals of group 1. Continue to add the HCl T.S. until the precipitation appears to be complete. Filter the liquid and test the clear filtrate by adding a few drops of HCl T.S. If this produces a precipitate add more HCl T.S., filter through the same filter paper and test the last portion of the filtrate in the same manner as before, and repeat the filtration and testing of the filtrate until the HCl T.S. produces no precipitate in the filtrate, when precipitation is complete. A small amount of lead, however, remains in the solution, if this metal is present.

B. Heat some water to boiling in a beaker supported on wire gauze over a gas flame. Pour 20 mls of this hot water through the precipitate on the filter paper. If lead is present, PbCl_2 dissolves. Test separate portions of the filtrate for lead with KI T.S., H_2S , H_2SO_4 T.S., and $\text{K}_2\text{Cr}_2\text{O}_7$ T.S. as described on page 17, remembering that the precipitates, if any, are small in amount because the solution of lead is very weak.

If lead is found pass 50 mls or more of boiling water through the precipitate on the filter paper to wash out the remainder of the PbCl_2 . When the PbCl_2 is all washed out a portion of the filtrate will not give a white precipitate with H_2SO_4 T.S., with which it should be tested.

If the precipitate all dissolves in hot water it consisted of PbCl_2 alone, and silver and mercurous mercury are absent.

C. If a precipitate remains on the filter paper it consists of AgCl or HgCl , or both. Pour a small amount of NH_4OH T.S. through the precipitate on the filter. If the precipitate blackens, the presence of mercurous mercury is shown, as described on page 25.

D. If silver is present the AgCl dissolves in the NH_4OH T.S. Add HNO_3 T.S. to the filtrate until it is acid in reaction to litmus. A white precipitate shows the presence of silver, as described on page 27.

GROUP 2.

Metals precipitated by hydrogen sulphide, H_2S , in presence of hydrochloric acid, HCl , and whose sulphides are insoluble in ammonium sulphide, $(\text{NH}_4)_2\text{S}_x$.

Mercuric mercury, Hg^{++} ; (lead, Pb); bismuth, Bi ; copper, Cu ; cadmium, Cd .

(RUM), $Hg^{ii} = 200.6$.

ous compounds to mer-
dily change mercuric com-
curic compounds are much
ds, care must be taken not
compound for medicinal use
to the mercuric condition.
ored, being white, yellow,
or only slightly soluble in
rate, and mercuric cyanide

COMPOUNDS.

um ammoniatum, U. S. P.,
ate," NH_2HgCl .

m corrosivum, U. S. P.,

um, U. S. P., "biniodide

n flavum, U. S. P., HgO .

n rubrum, U. S. P., "red

" $(HgO)_2HgSO_4$.

el," HgS .

emilion," HgS .

MERCURY.

a mercuric salt, as mercuric
tests:

ution. A black precipitate
several other colors, rang-
nd brown, generally appear
ange of colors is due to the
 HgS with the original mer-

solution. An excess of the
 HgO . When the mercuric
n and consists of a basic
obtained with lime water,
the old fashioned "yellow

3. Add NH_4OH T.S. to the mercuric solution. A white precipitate of mercurammonium chloride, NH_2HgCl , is formed. Add HCl T.S. The precipitate will dissolve.

4. Slowly add KI T.S. to the mercuric solution. A precipitate of mercuric iodide, HgI_2 , first yellow, then red, is produced, which forms potassium-mercuric iodide, $(\text{KI})_2\text{HgI}_2$, and dissolves in an excess of the reagent.

5. Slowly add SnCl_2 T.S. to the mercuric solution. At first the precipitate formed is white and consists of mercurous chloride, HgCl . When the reagent is in excess, the precipitate is gray and consists of finely divided metallic mercury.

6. Acidulate a small amount of the mercuric solution in a small beaker with HCl T.S. and introduce a small piece of copper foil into the liquid. Mercury is deposited on the copper, just as was described for mercurous compounds on page 18.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{HgCl}_2 + \text{H}_2\text{S} =$
 $\text{Hg}(\text{NO}_3)_2 + (\text{NH}_4)_2\text{S} =$
2. $\text{HgCl}_2 + \text{NaOH} =$
 $\text{HgCl}_2 + \text{Ca}(\text{OH})_2 =$
3. $\text{HgCl}_2 + \text{NH}_4\text{OH} = \text{NH}_2\text{HgCl} +$
 $\text{NH}_2\text{HgCl} + \text{HCl} =$
4. $\text{HgCl}_2 + \text{KI} =$
 $\text{Hg}(\text{NO}_3)_2 + \text{NH}_4\text{I} =$
5. $\text{HgCl}_2 + \text{SnCl}_2 =$
 $\text{HgCl} + \text{SnCl}_2 =$
6. $\text{HgCl}_2 + \text{Cu} =$

BISMUTH, $\text{Bi}^{\text{iii}}, \nu = 208.0$.

Bismuth is a hard and brittle crystalline metal which is a constituent of some easily fusible alloys and is used in manufacturing its compounds. The salts of bismuth are generally white in color, but the oxide is yellow and the sulphide is black. Bismuth chloride and bismuth nitrate are soluble in water with some decomposition. The other important compounds are insoluble. Bismuth compounds in solution readily form insoluble basic compounds containing the monad radical *bismuthyl*, $(\text{BiO})'$. Some organic acids or large amounts of mineral acids prevent this precipitation. Bismuthic compounds are unimportant.

IMPORTANT COMPOUNDS OF BISMUTH.

Bismuth chloride, BiCl_3 .

Bismuth subcarbonate, U. S. P., mainly $(\text{BiO})_2\text{CO}_3 \cdot \text{H}_2\text{O}$.

Bismuth nitrate, $\text{Bi}(\text{NO}_3)_3$.

Bismuth oxide, Bi_2O_3 .

Bismuth subnitrate, U. S. P., mainly, $\text{Bi}(\text{OH})_2\text{NO}_3$.

TH.

of either bismuth nitrate, each of the following tests: on into 10 mils of water ate of bismuth subnitrate, is produced if the liquid

tion. A black precipitate

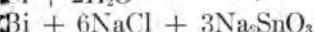
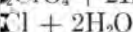
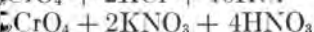
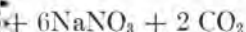
solution. A white precipi- formed. Boil the liquid ate changes to yellow bis- re present the precipitate illic bismuth.

solution. A white precipi- is produced.

solution. A yellow pre- CrO_4 is formed, which is aromates, as K_2CrO_4 , give

SnCl_2 T. S. until the pre- odium stannite, Na_2SnO_2 . to the Na_2SnO_2 solution. i, is produced.

LOWING EQUATIONS:



= 63.57

used in the metallic state, composed of copper and

zinc, is the most important. Many of the compounds of copper are green or blue in color and they impart these colors to their solutions. The cuprous compounds are unstable and unimportant. Copper sulphate, copper chloride and copper nitrate are readily soluble in water. The other important compounds of copper are insoluble or only slightly soluble in water.

IMPORTANT COMPOUNDS OF COPPER.

Basic copper acetate, "verdigris," $\text{Cu}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$.

Copper aceto-arsenite, "Paris green," $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2 + 3\text{Cu}(\text{AsO}_2)_2$.

Copper arsenite, "Scheele's green," CuHAsO_3 .

Copper chloride, cupric chloride, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$.

Copper nitrate, cupric nitrate, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$.

Cuprous oxide, Cu_2O .

Cupric oxide, CuO .

Copper sulphate, cupric sulphate, *Cupri sulphas*, U. S. P., "blue vitriol," $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

TESTS FOR COPPER.

A separate portion of a solution of a copper salt, as copper sulphate, CuSO_4 , should be used for each of the following tests:

1. Pass H_2S through the copper solution. A black precipitate of copper sulphide, CuS , is formed. This precipitate is soluble in nitric acid and in a solution of potassium cyanide.

2. Add NaOH T.S. to the copper solution. A light blue precipitate of copper hydroxide, $\text{Cu}(\text{OH})_2$, is produced, which is insoluble in an excess of the reagent. Boil the liquid containing the precipitate. The copper hydroxide is decomposed into black cupric oxide, CuO , and water.

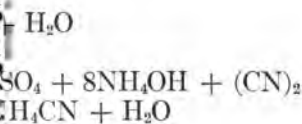
If the liquid contains citrates or tartrates or certain other organic substances, the copper hydroxide will dissolve in an excess of the alkali, forming a deep blue solution from which red cuprous oxide, Cu_2O will precipitate on boiling if reducing agents are present. This behavior is the basis of Fehling's test for sugar and other similar tests.

3. Slowly add NH_4OH T.S. to the solution of a copper salt. A light blue precipitate of copper hydroxide, $\text{Cu}(\text{OH})_2$, is produced and immediately dissolves in an excess of the reagent, forming a deep blue solution containing ammonio-copper sulphate, $\text{Cu}(\text{NH}_3)_4\text{SO}_4$, or a similar compound. Slowly add KCN T.S. to the blue liquid. Potassium cuprocyanide, $\text{K}_3\text{Cu}(\text{CN})_4$, is formed and the liquid becomes colorless. H_2S does not precipitate the copper from this solution, as copper sulphides are soluble in a solution of KCN .

4. Add $\text{K}_4\text{Fe}(\text{CN})_6$ T.S. to the solution of a copper salt. A red-brown precipitate of copper ferrocyanide, $\text{Cu}_2\text{Fe}(\text{CN})_6$, is produced, which is insoluble in diluted acids.

copper solution and heat
The flame will be colored

LOWING EQUATIONS:



12.4.

tal resembling zinc and tin.
alloys. Cadmium salts are
is yellow and the oxide is
e halogen salts are soluble
alts are insoluble in water.

OF CADMIUM.

UM.

a cadmium salt, as CdSO_4 ,
olution. A yellow precipi-
This precipitate is soluble
by other diluted acids, nor
des.
solution. A white precipi-
all form, which is insoluble

in an excess of the reagent. The same precipitate will form with NH_4OH T.S., but is soluble in an excess of this reagent.

3. Add Na_2CO_3 T.S. to the cadmium solution. A white precipitate of cadmium carbonate, CdCO_3 , is formed, which is not soluble in an excess of the reagent, but which will dissolve in diluted acids.

4. Slowly add KCN T.S. to the cadmium solution. A white precipitate of cadmium cyanide, $\text{Cd}(\text{CN})_2$, will be produced and this will form potassium cadmium cyanide, $\text{K}_2\text{Cd}(\text{CN})_4$, and dissolve in an excess of the reagent. Pass H_2S through the solution. Yellow cadmium sulphide, CdS , will be precipitated.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{CdSO}_4 + \text{H}_2\text{S} =$
 $\text{Cd}(\text{NO}_3)_2 + \text{H}_2\text{S} =$
2. $\text{CdSO}_4 + \text{NaOH} =$
 $\text{Cd}(\text{NO}_3)_2 + \text{NH}_4\text{OH} =$
3. $\text{Cd}(\text{NO}_3)_2 + \text{Na}_2\text{CO}_3 =$
 $\text{CdSO}_4 + (\text{NH}_4)_2\text{CO}_3 =$
4. $\text{CdSO}_4 + \text{KCN} =$
 $\text{Cd}(\text{CN})_2 + \text{KCN} =$
 $\text{K}_2\text{Cd}(\text{CN})_4 + \text{H}_2\text{S} =$

SAMPLE NUMBER 2.

Analysis of a solution for group 2 of the metals.

The solution may contain salts of mercuric mercury, lead, bismuth, copper or cadmium, or of several of these metals.

Add a few drops of HCl T.S. to about 10 mls of the sample, warm and pass H_2S through the acidulated liquid. If no precipitate is produced, the metals of group 2 are absent. If a precipitate is produced, continue to pass H_2S through the liquid until precipitation is complete.

Collect the precipitate on a filter and test the filtrate by passing H_2S through it for a few minutes. If any additional precipitate is formed, continue to pass H_2S through the liquid for some time and then filter through the same filter paper, testing the new filtrate in the same manner as before. The metals of this group are all in the precipitate on the filter paper and the filtrate may be discarded, unless it is to be tested for the metals of subsequent groups.

A. Wash the precipitate on the filter with water and allow the water to drain out, discarding the washings. Pour the same portion of about 5 mls of hot HNO_3 T.S. through the filter several times. Any white or light colored residue insoluble in HNO_3 T.S. may be disregarded. A dark colored residue, insoluble in HNO_3 T.S. should

filtrate is to be tested for the

that will not dissolve in
by puncturing the filter
tube with about 3 mils of
til it ceases to smell of
filter and add SnCl_2 T.S.

ence of mercury.

a beaker, add about 2
white fumes are given off.
r. A white precipitate
r and save the filtrate to
sub-group.

in the precipitate on the
drain out and pass about
per several times. Add
precipitate shows the presence

an excess of NH_4OH T.S.,
persists after shaking the
presence of bismuth. If a
reserve the filtrate to be

wash the precipitate on
in and moisten it with a
drops of SnCl_2 T.S. A
e of bismuth.

blue, copper is present.
by adding an excess of
S. to a portion of the
shows the presence of

orless, pass H_2S through
nce of cadmium. If the
portion, until the blue
ch the liquid. A yellow
n.

ES.

analytical processes are
ch tables are very con-
ough, however, to be fol-
s, so they should be used

only by those who have become familiar with the processes by following detailed directions.

In using the tables in this book the following points should be kept in mind, constantly:

Where chemical formulas are given for reagents, the test solutions should be used, unless directed otherwise.

Care should be taken to use sufficient of each reagent to accomplish its purpose, without using a large excess, and to test all filtrates after removing a metal or group by precipitation.

Only positive results are given in the tables, as if all of the metals included are present. When a negative result is obtained on testing for any metal or group, it is absent and one should pass on to the next test, often omitting filtrations or other processes which would be necessary if positive results were obtained.

SAMPLE NUMBER 3.

Analysis of a solution for groups 1 and 2 of the metals.

Examine the solution by the following tables, referring when necessary to the detailed directions for each group given on preceding pages.

The solution should be neutral or acid to litmus. If alkaline, add HNO_3 to the portion to be examined until the reaction is acid.

Add HCl as long as a precipitate is produced and filter.

Precipitate— Pb , Hg^{I} , Ag

Examine for metals of group 1 by I.

Filtrate— Hg^{II} , Pb , Bi , Cu , Cd

Warm and pass H_2S as long as a ppt. is produced. Filter. Examine for metals of group 2 by II.

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

Filtrate— PbCl_2

Add $\text{K}_2\text{Cr}_2\text{O}_7$ and cool.
Yellow ppt., PbCrO_4 ,
shows

lead

Residue— HgCl , AgCl

Pass the same portion of NH_4OH through the residue on the filter several times

Residue— $\text{NH}_2\text{Hg}_2\text{Cl}$
black, shows

mercurous mercury

Filtrate— $(\text{NH}_4)_2(\text{AgCl})_2$
Add an excess of HNO_3 ,
white ppt., AgCl , shows

silver

by H_2S in an acid liquid, from a solution.

...n, discarding the washings. several times.

... $\text{Cu}(\text{NO}_3)_2$, $\text{Cd}(\text{NO}_3)_2$

...fumes are given off, dilute and filter.

... $(\text{SO}_4)_2$, CuSO_4 , CdSO_4

...of NH_4OH and filter.

Filtrate—

... $(\text{NH}_4)_2\text{SO}_4$, $\text{Cd}(\text{NH}_4)_2\text{SO}_4$

Divide into two portions.

...one portion
...an excess
... $\text{HC}_2\text{H}_3\text{O}_2$
...and then
... $\text{Fe}(\text{CN})_6$.
...red ppt. or
...oloration,
... $\text{Fe}(\text{CN})_6$,
...shows

copper

Pass H_2S through the other portion, first decolorizing with KCN if Cu is present. A yellow ppt., CdS , shows

cadmium

hydrogen sulphide, H_2S , and whose sulphides are

...imony, Sb ; Tin, Sn .

197.2.

...ost largely in the metallic
...t in the air and does not
...d by nitro-hydrochloric
...rine, bromine or iodine.
...es and fused potassium
...nd unstable. The only
...e salts with the halogen
...brown or yellow in color.

IMPORTANT COMPOUNDS OF GOLD.

Gold chloride, AuCl_3 .

Gold and sodium chloride, *Auri et Sodii Chloridum*, U. S. P., $\text{AuCl}_3 + \text{NaCl}$.

Bromauric acid, N. F., $\text{HAuBr}_4 + 5\text{H}_2\text{O}$.

Chlorauric acid, $\text{HAuCl}_4 + 4\text{H}_2\text{O}$.

TESTS FOR GOLD.

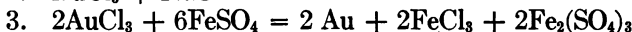
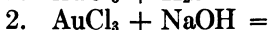
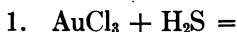
Use a separate portion of a solution of gold chloride, AuCl_3 , for each of the following tests:

1. Pass H_2S gas through the gold solution, obtaining a brownish-black precipitate of gold sulphide, Au_2S_3 . Collect the precipitate on a filter and pass yellow ammonium sulphide solution through the filter. The precipitate will dissolve.

2. Add NaOH T.S. A brown precipitate of gold hydroxide, $\text{Au}(\text{OH})_3$, soluble in an excess of the reagent will be formed.

3. Add 2 drops of nitric acid and then FeSO_4 T.S. A brown precipitate of metallic gold will be formed.

4. Add SnCl_2 T.S. and let stand. A purple precipitate of "Purple of Cassius" is slowly formed. This is a substance of indefinite composition, containing aurous oxide, Au_2O , and oxides of tin.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:**PLATINUM, $\text{Pt}^{\text{ii, iv}} = 195.2$.**

Platinum is a heavy white metal with a high melting-point. It is permanent in the air and is not attacked by any single acid. Platinum vessels are used in chemical work and care should be taken not to allow them to come in contact with free chlorine or bromine, nor to ignite alkalies in them, nor mixtures that contain free iodine, sulphur, phosphorus or metals, or which will liberate these substances. The reducing gas flame forms a platinum carbide, so care should be taken to heat platinum vessels only in the oxidizing flame. Platinum ware may be cleansed by fusing borax in it and it may be polished by rubbing with moist sea-sand.

Platinum salts are unstable and readily reduced, liberating the metal as a black porous mass called "spongy platinum" or a black powder called "platinum black."

IMPORTANT COMPOUNDS OF PLATINUM.

Platinum chloride, $\text{PtCl}_4 + 5\text{H}_2\text{O}$.

Chloroplatinic acid, $\text{H}_2\text{PtCl}_6 + 6\text{H}_2\text{O}$.

NUM.

platinum chloride, PtCl_4 ,

solution. A black precipitate slowly in a cold liquid, insoluble in acids, but is

volume of alcohol. A yellow $(\text{NH}_4)_2\text{PtCl}_6$ is formed.

ate of the corresponding

LOWING EQUATIONS:

74.96.

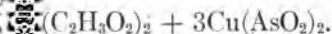
at which has the physical
aves as a non-metal. The
sons and methods for the
ent in the stomach contents
great importance in toxico-

ally white, though several
ne other elements present.
enic are more or less soluble
reen are insoluble. Com-
gh temperatures, with or

OF ARSENIC.



arsenous anhydride, "white



n, "arsine," AsH_3 .



NIC.

f either arsenous chloride,
lium arsenate, Na_2HASO_4 ;

1. Pass H_2S gas through the warmed arsenic solution. A yellow precipitate of arsenous sulphide, As_2S_3 , is formed, quickly from strong solutions of arsenous compounds and slowly from weak solutions of arsenous compounds or from solutions of arsenic compounds.

Collect the precipitate on a filter paper and pass warm $(\text{NH}_4)_2\text{S}$ T.S. through the paper. The precipitate dissolves.

Arsenous sulphide is also dissolved by $(\text{NH}_4)_2\text{CO}_3$ T.S.

2. REINSCH'S TEST.—Mix 20 mls of HCl T.S. with 20 mls of water and heat to boiling in a small porcelain evaporating dish, supported on a piece of wire gauze on a tripod over the flame of a Bunsen burner. When the liquid boils introduce a piece of bright copper foil about 2 cm. square and continue boiling for five minutes, adding water from time to time to keep the volume of the liquid approximately constant. If there is no dark deposit formed on the copper, the reagent and apparatus are free from arsenic.

A dark deposit on the copper shows the presence of arsenic or other easily reduced metal in the reagents. Such a preliminary trial is called a *blank test*, and is made when there is a possibility of misleading results being obtained from the reagents or other source.

If the copper remains bright during the preliminary test, add two or three drops of the arsenic solution and boil for ten minutes, adding water from time to time to keep the volume constant. A dark coating of copper arsenide, Cu_3As_2 , will be deposited on the copper, which may scale off if much arsenic is present. Pour off the liquid, rinse the copper, first with water and then with alcohol, and then dry it by pressing between the folds of a filter paper. Cut the copper into small pieces, introduce some of them into a dry test-tube, and heat the lower end of the tube by holding it in the flame of a Bunsen burner. The arsenic will oxidize and volatilize, condensing on the cool part of the tube as a white deposit of arsenic trioxide, As_2O_3 , whose characteristic octahedral crystals may be recognized under a magnifying glass.

Reinsch's test will detect very small amounts of arsenic, and it can be used in the presence of organic matter.

3. MARSH'S TEST.—Prepare a small hydrogen generator with an outlet tube drawn to a jet. Introduce a few pieces of pure zinc and some diluted hydrochloric acid into the generator and allow the generated hydrogen to escape for some time, to displace all of the air in the generator. To test this hold an inverted test-tube down over the jet until the tube is filled with hydrogen. Remove it from the jet, keeping the test-tube inverted, and bring the mouth of the tube in contact with a small flame. If the hydrogen burns quietly at the mouth of the tube it is safe to ignite it at the jet, but not otherwise. To make it doubly sure wrap a towel loosely

is quietly in the test-tube, the hydrogen. The towel of porcelain in the flame discolored the reagents and a dark spot appears on the plain arsenic or antimony.

The apparatus to be free of arsenic and introduce a few drops of water through the funnel. A small part of the nascent arsine, AsH_3 , is formed. In a short time the flame, the arsine burning to a piece of cold porcelain, the nature of the burning gas, deposited on the porcelain. Chlorinated soda, NaClO .

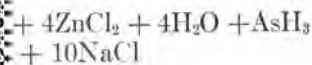
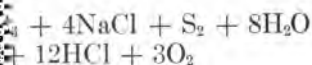
It is if a mixture containing the organic matter must be

added to a solution of Na_2HAsO_4 . Magnesium arsenate, $\text{MgNH}_4\text{AsO}_4$,

Na_2HAsO_3 . No precipi-

precipitates of arsenous and arsenic

THE FOLLOWING EQUATIONS:



$$= 120.2.$$

all whose compounds are antimony and lead. Soluble in solutions that con-

tain moderate amounts of mineral acids and basic compounds containing the monad radical *antimonyl*, $(\text{SbO})'$, or more complex oxides, are precipitated. Organic acids or large amounts of mineral acids prevent this precipitation.

Antimony salts are unstable and only a few of them are important. The oxides are feebly basic with acids and feebly acid with bases. Antimonous chloride and the other halogen salts are deliquescent and soluble in small amounts of water with some decomposition. Antimonyl-potassium tartrate is moderately soluble in water, without decomposition.

IMPORTANT COMPOUNDS OF ANTIMONY.

Antimonous chloride, "butter of antimony," SbCl_3 .

Antimonous oxide, Sb_2O_3 .

Antimonous sulphide, Sb_2S_3 .

Antimonyl-potassium tartrate, *Antimony and potassium tartrate*, U. S. P., "tartar emetic," $(\text{KSbOC}_4\text{H}_4\text{O}_6)_2\text{H}_2\text{O}$.

Hydrogen antimonide, "stibine," SbH_3 .

TESTS FOR ANTIMONY.

Use separate portions of a solution of antimonous chloride, SbCl_3 , for each of the following tests:

1. Pour 2 mls of the antimony chloride solution into a test-tube half filled with water. A white precipitate consisting mainly of antimony oxychloride, SbOCl , is formed if the solution does not contain a large excess of free acid.

2. Pass H_2S gas through the antimony solution. An orange red precipitate of antimony sulphide, Sb_2S_3 , is formed. Collect a small amount of the precipitate on a filter and pass warm yellow ammonium sulphide solution through the filter. The precipitate dissolves.

3. Apply Reinsch's test, including the blank test, as described under arsenic on page 4, but using a few drops of the antimony solution instead of the arsenic solution. A dark gray deposit of metallic antimony will form on the copper. If the copper with the deposit is dried and cut into small pieces and heated in a dry test-tube the antimony will oxidize and volatilize, condensing on the cool part of the tube as a white deposit of antimonous oxide, Sb_2O_3 , which is amorphous.

4. Apply Marsh's test, including the blank test, as described under arsenic on pages 40-41, but using a few drops of the antimony solution, instead of the arsenic solution. The antimony compound is reduced by a small part of the nascent hydrogen and gaseous stibine, SbH_3 , is formed, which burns with the excess of hydrogen

water. If cold porcelain is deposited on it, forming dull, solution of chlorinated soda. into AgNO_3 T.S. If stibine antimonide, Ag_3Sb , is formed. on a filter and wash well per the test-tube and pass if necessary to get a clear Cl_2 and the silver remains the filtrate and an orange

LOWING EQUATIONS:

$$= 119.0.$$

that is largely used in the various alloys like bronze, y called "tin," is sheet iron

DS OF TIN.

stannous chloride, SnCl_2 ,

chloride solution into about 10 as oxychloride, Sn_2OCl_2 , will acid.

precipitates stannous oxychloride

with formation of some stannic chloride in bottles of SnCl_2 T.S. This precipitation is retarded by acidulating the solution with HCl , and keeping some pieces of metallic tin in it.

2. Pass H_2S through the SnCl_2 solution. A brown precipitate of stannous sulphide, SnS , is formed. Collect a small amount of the precipitate on a filter and pass yellow ammonium sulphide solution through the filter. The precipitate will dissolve, forming ammonium sulphostannate, $(\text{NH}_4)_2\text{SnS}_3$. Add an excess of diluted HCl to the filtrate. A yellow precipitate of stannic sulphide, SnS_2 , will form.

3. Add NaOH T.S. in excess. A white precipitate of stannous hydroxide, $\text{Sn}(\text{OH})_2$, appears and then dissolves in the excess of the reagent, forming sodium stannite, Na_2SnO_2 . Boil the solution gently. A white precipitate of stannous oxide, SnO , forms.

4. Add a few drops of diluted HCl and a few drops of HgCl_2 T.S. to the solution of SnCl_2 . A white precipitate of mercurous chloride, HgCl , will form, or, if the SnCl_2 is in large excess, the precipitate will be gray and consist of metallic mercury.

5. Pour some SnCl_2 solution into a test-tube containing zinc and diluted HCl . Metallic tin is liberated as a gray, spongy precipitate which will form SnCl_2 and dissolve in HCl if the pieces of zinc are removed.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{SnCl}_2 + \text{H}_2\text{O} =$
2. $\text{SnCl}_2 + \text{H}_2\text{S} =$
 $\text{SnS} + (\text{NH}_4)_2\text{S}_2 =$
 $(\text{NH}_4)_2\text{SnS}_3 + \text{HCl} = \text{SnS}_2 +$
3. $\text{SnCl}_2 + \text{NaOH} =$
 $\text{Sn}(\text{OH})_2 + \text{NaOH} =$
 $\text{Na}_2\text{SnO}_2 + \text{H}_2\text{O} =$
4. $\text{SnCl}_2 + \text{HgCl}_2 = \text{HgCl} +$
 $\text{SnCl}_2 + \text{HgCl}_2 = \text{Hg} +$
5. $\text{SnCl}_2 + \text{Zn} =$
 $\text{Sn} + \text{HCl} =$

NOTE ON STANNIC COMPOUNDS.

Stannic compounds in solution give a yellow precipitate of stannic sulphide, SnS_2 , with H_2S , and a white precipitate of stannic hydroxide, $\text{Sn}(\text{OH})_4$, with NaOH T.S., soluble in an excess of the reagent. Lead and hydrochloric acid reduce stannic compounds, SnCl_2 being formed, which may be recognized by applying tests for stannous compounds to the solution.

SAMPLE NUMBER 4.

Analysis of a solution for group 3 of the metals.

The solution may contain salts of arsenic, antimony, tin, gold or platinum, or of several of these metals.

with HCl T.S., warm and

passing H_2S through the acid
soluble amounts of the metals

pass H_2S through the liquid
complete.

test the filtrate by passing
more precipitate is formed,
it may be discarded, unless
groups. If H_2S produces
pass the gas through the
through the same filter paper,
in the same manner as before.

the precipitate on the filter
and wash the precipitate into
T.S., which will dissolve
other metals of this group.
tube in hot water for a few
on the filter with 10 mils of

with HCl T.S. and pass H_2S
the presence of arsenic. A
and may be disregarded. If
Reinsch's test to the liquid

to drain on the filter paper.
the residue into a test-tube
at the test-tube by standing
over. The filtrate will contain
the insoluble residue will con-

to a small generating flask
tube. Add an equal volume
of dilute zinc. Conduct the gas
with $AgNO_3$ T.S. for three minutes.
T.S., antimony is absent. A
of antimony.

in the $AgNO_3$ T.S. by para-
filter and wash with water,
then pass HCl T.S. through
the filter again if necessary
through the filtrate. An orange-
of antimony.

in the generator, described

in paragraph *E*, through a filter into about 1 mil of HgCl_2 T.S. contained in a test-tube. A white or gray precipitate shows the presence of tin.

H. If the residue, insoluble in hydrochloric acid is light colored the sample does not contain gold or platinum and the residue may be discarded. If this residue is dark colored it should be tested for gold and platinum. Puncture the apex of the filter paper and wash the residue into a test-tube with 5 mils of nitrohydrochloric acid. Warm the liquid by standing the test-tube in hot water, add an equal volume of water, filter out any insoluble residue and divide the clear filtrate into two parts.

I. To one part of the filtrate add SnCl_2 and SnCl_4 test solutions and let stand. A purple coloration or precipitate indicates gold. Verify by adding SnCl_2 and SnCl_4 test solutions to the original solution.

J. Neutralize the other part of the filtrate from *H* with NH_4OH , add an equal volume of alcohol and then NH_4Cl T.S. A yellow or brown precipitate indicates platinum. Verify by adding alcohol and NH_4Cl T.S. to the original solution.

SAMPLE NUMBER 5.

Analysis of a solution for groups 1, 2 and 3 of the metals.

The solution should be neutral or acid in reaction. If alkaline, add HNO_3 to the portion to be examined until the reaction is acid.

Add HCl as long as a precipitate is produced and filter.

Precipitate—Pb, Hgⁱ, Ag

Examine for metals of group 1 by I.

Filtrate—

Warm and pass H_2S as long as a precipitate is produced. Collect the ppt. on a filter, wash and drain. Digest the ppt. with $(\text{NH}_4)_2\text{S}_x$ and filter.

Residue—

Hgⁱⁱ, Pb, Bi, Cu, Cd

Examine for metals of group 2 by II.

Filtrate—

As, Sb, Sn, Au, Pt

Examine for metals of group 3 by III.

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

Filtrate—PbCl₂

Add K_2CrO_4 . Yellow ppt., PbCrO_4 , shows

lead

Residue—HgCl, AgCl

Pass the same portion of NH_4OH through the residue on the filter several times

Residue— $\text{NH}_2\text{HgCl} + \text{Hg}$, black, shows

mercurous mercury

Filtrate— $(\text{NH}_4)_2(\text{AgCl})_2$. Add an excess of HNO_3 , white ppt., AgCl , shows silver

$(\text{NH}_4)_2\text{S}_x$ from a precipitate group 1 of the metals

Portion of $(\text{NH}_4)_2\text{S}_x$ and

several times.

$\text{Cu}(\text{NO}_3)_2$, $\text{Cd}(\text{NO}_3)_2$

white fumes are given off, cool, and filter.

$(\text{SO}_4)_2$, CuSO_4 , CdSO_4

of NH_4OH and filter.

Filtrate—

$\text{Cu}(\text{NH}_3)_4\text{SO}_4$, $\text{Cd}(\text{NH}_3)_4\text{SO}_4$

Divide into two portions.

one portion

and an excess

$\text{H}_2\text{C}_2\text{O}_4$

and then

$\text{K}_4\text{Fe}(\text{CN})_6$

red ppt. or

coloration,

$\text{Cu}_2\text{Fe}(\text{CN})_6$,
shows

copper

Pass H_2S

through the

other portion,

decolorizing

with KCN , if

Cu is present.

A yellow ppt.,

CdS , shows

cadmium

of any precipitate group 1 of the metals has been

collect any precipitate formed and digest in $(\text{NH}_4)_2\text{CO}_3$ T.S.

Au_2S_3 , PtS_2

concentrated HCl . Filter.

Residue— Au_2S_3 , PtS_2

light colored Au and Pt

absent. If dark colored, digest in nitrohydrochloric acid.

add H_2O and filter.

a part of

the filtrate add

SnCl_2 and

HCl , and let

stand. A pur-

ple ppt., Au_2O_3 ,
etc., shows

gold

Neutralize

another part of

the filtrate with

NH_4OH , add

alcohol and

NH_4Cl . A yellow ppt.,

$(\text{NH}_4)_2\text{PtCl}_6$,
shows

platinum

GROUP 4.

Metals precipitated as hydroxides by ammonium hydroxide, NH_4OH , in presence of ammonium chloride, NH_4Cl :

Iron, Fe; chromium, Cr; aluminum, Al.

IRON (FERRUM) $\text{Fe}^{\text{ii, iii}}$ = 55.84

Iron is largely used in the metallic state, nearly pure as *wrought iron*, less pure as *steel*, and still less pure as *cast iron* and *malleable iron*. *Reduced iron* is powdered metallic iron obtained by the action of hydrogen on heated ferric oxide.

In the ferrous compounds, iron has a valence of two, and in the ferric compounds it has a valence of three. Ferrous compounds are readily oxidized to ferric compounds and ferric compounds are nearly as readily reduced to ferrous compounds. Both series of compounds are important. Soluble ferrous compounds are generally light green in color and slightly color their solutions. Soluble ferric compounds are generally brown or yellow in color and strongly color their solutions.

IMPORTANT FERROUS COMPOUNDS.

Ferrous bromide, $\text{FeBr}_2 \cdot 6\text{H}_2\text{O}$.

Ferrous carbonate, FeCO_3 .

Ferrous chloride, FeCl_2 .

Ferrous hydroxide, $\text{Fe}(\text{OH})_2$.

Ferrous iodide, FeI_2 .

Ferrous oxide, FeO .

Ferrous sulphate, U. S. P., "copperas," "green vitriol," $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

Exsiccated ferrous sulphate, U. S. P., $(\text{FeSO}_4)_2 \cdot 3\text{H}_2\text{O}$.

Ferrous sulphide, FeS .

IMPORTANT FERRIC COMPOUNDS.

Ferric ammonium sulphate, "ferric alum," $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Ferric chloride, U. S. P., $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

Ferric ferrocyanide, "Prussian blue," $\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$.

Ferric hydroxide, "arsenic antidote," $\text{Fe}(\text{OH})_3$.

Ferric hypophosphite, N. F., $\text{Fe}(\text{PH}_2\text{O}_2)_3$.

Ferric nitrate, $\text{Fe}(\text{NO}_3)_3$.

Ferric oxide, "hematite," Fe_2O_3 .

Ferroso-ferric oxide, "magnetite," Fe_3O_4 .

Ferric phosphate, U. S. P., composition indefinite.

Ferric pyrophosphate, N. F., composition indefinite.

Ferric sulphate, $\text{Fe}_2(\text{SO}_4)_3$.

Ferric subsulphate, "Monsel's salt," $\text{Fe}_4\text{O}(\text{SO}_4)_5$.

IRON.

a ferrous salt, as FeSO_4 , for

solution. A white precipitate is produced if the ferrous salt is present. It begins to oxidize and darken to ferric hydroxide, $\text{Fe}_2(\text{OH})_6$, on exposure to the light brown precipitate. Ferric hydroxide is slightly soluble in water. Precipitation of iron by this reagent will produce the same results. In acid solution. A white precipitate is produced and immediately begins to darken in color, forming first the ferric hydroxide, $\text{Fe}_2(\text{OH})_6$, and then, by further oxidation, $\text{Fe}(\text{OH})_3$. Na_2CO_3

solution. A black precipitate is produced. The precipitate is

insoluble in acid solution. A light blue precipitate, $\text{K}_2\text{FeFe}(\text{CN})_6$ will be formed. Ferric ferrocyanide, $\text{Fe}_3(\text{Fe}(\text{CN})_6)_2$, is insoluble in acids. In acid solution the precipitate

is insoluble in acid solution. A dark blue precipitate, $\text{Fe}(\text{CN})_6^{4-}$, will be formed.

solution. No coloration or precipitate is formed if some ferric salt is present, as the reagent.

Reactions:

TESTS FOR FERRIC IRON.

Use a separate portion of a solution of a ferric salt, as FeCl_3 , for each of the following tests:

1. Add NH_4OH T.S. to the ferric solution. A red-brown gelatinous precipitate of ferric hydroxide, $\text{Fe}(\text{OH})_3$, will be formed, which is insoluble in an excess of the reagent or in solutions of ammonium salts, but which will dissolve in acids. Precipitation is not complete if glycerine, sugar, or some organic acids are present. On boiling the liquid containing the precipitate, it changes to a basic ferric hydroxide, $\text{FeO}(\text{OH})$, which is not gelatinous.

NaOH T.S. produces the same precipitate.

2. Add Na_2CO_3 T.S. to the ferric solution. Carbon dioxide CO_2 , is given off, and ferric hydroxide, $\text{Fe}(\text{OH})_3$, is precipitated. The same result is obtained with $(\text{NH}_4)_2\text{CO}_3$ T.S.

3. Add $(\text{NH}_4)_2\text{S}$ T.S. to the ferric solution. The iron is reduced to the ferrous condition and a black precipitate is obtained, which consists of ferrous sulphide, FeS , and sulphur.

H_2S reduces ferric salts in solution to the ferrous condition, with precipitation of sulphur.

4. Add $\text{K}_4\text{Fe}(\text{CN})_6$ T.S. to the ferric solution. A deep blue precipitate of ferric ferrocyanide, $\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$, is formed. The precipitate is insoluble in diluted acids, but is decomposed by alkalis.

5. Add $\text{K}_3\text{Fe}(\text{CN})_6$ T.S. to the ferric solution. Ferric ferricyanide, $\text{FeFe}(\text{CN})_6$, is produced and changes the color of the solution to a greenish-brown.

6. Add KCNS T.S. to the ferric solution. A blood red coloration is produced, due to the formation of ferric sulphocyanate, $\text{Fe}(\text{CNS})_3$. This reaction is most dependable in the presence of moderate amounts of free HCl . Many different compounds prevent the reaction in neutral solutions.

Complete and balance the following equations:

1. $\text{FeCl}_3 + \text{NH}_4\text{OH} =$
 $\text{Fe}_2(\text{SO}_4)_3 + \text{NaOH} =$
 $\text{Fe}(\text{OH})_3 + \text{heat} =$
2. $\text{Fe}(\text{NO}_3)_3 + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} =$
 $\text{FeCl}_3 + (\text{NH}_4)_2\text{CO}_3 + \text{H}_2\text{O} =$
3. $\text{Fe}_2(\text{SO}_4)_3 + 3(\text{NH}_4)_2\text{S} = 2\text{FeS} + \text{S} + 3(\text{NH}_4)_2\text{SO}_4$
 $\text{Fe}(\text{NO}_3)_3 + \text{K}_2\text{S} =$
 $\text{FeCl}_3 + \text{H}_2\text{S} =$
4. $\text{FeCl}_3 + \text{K}_4\text{Fe}(\text{CN})_6 =$
 $\text{Fe}_2(\text{SO}_4)_3 + \text{K}_4\text{Fe}(\text{CN})_6 =$
5. $\text{Fe}(\text{NO}_3)_3 + \text{K}_3\text{Fe}(\text{CN})_6 =$
6. $\text{FeCl}_3 + \text{KCNS} =$
 $\text{Fe}_2(\text{SO}_4)_3 + \text{KCNS} =$

52.0

which is seldom seen in
 plants in some varieties of

colored. When it has a
 important chromous com-
 forms the chromic com-
 it is an acid-forming
 and the chromates.

CHROMIUM.

hydride, "chromic acid,"

um," $\text{KCr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

CHROMIC SALTS.

chromic salt, as $\text{Cr}_2(\text{SO}_4)_3$,

solution. A greenish-blue
 $\text{Cr}(\text{OH})_3$, will form.

chromium solution. The
 will form and re-dissolve
 e, NaCrO_2 . Chromium

solution. The greenish-
 precipitated and carbon

solution. The greenish-
 precipitated and H_2S

from any of the preced-
 to drain. Transfer a
 e, add about an equal
 Na_2CO_3 . Support the
 e Bunsen burner flame
 the chromium to chro-
 and sodium chromates,
 and dissolve from the

cover by boiling with a small quantity of water in a beaker. Filter the solution formed, acidulate the filtrate with $\text{HC}_2\text{H}_3\text{O}_2$ and add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. A yellow precipitate of lead chromate, PbCrO_4 , will be formed.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Cr}_2(\text{SO}_4)_3 + \text{NH}_4\text{OH} =$
 $\text{CrCl}_3 + \text{NH}_4\text{OH} =$
2. $\text{Cr}_2(\text{SO}_4)_3 + \text{NaOH} =$
 $\text{Cr}(\text{OH})_3 + \text{NaOH} =$
 $\text{NaCrO}_2 + \text{H}_2\text{O} =$
3. $\text{CrCl}_3 + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} =$
 $\text{Cr}_2(\text{SO}_4)_3 + (\text{NH}_4)_2\text{CO}_3 + \text{H}_2\text{O} =$
4. $\text{Cr}_2(\text{SO}_4)_3 + (\text{NH}_4)_2\text{S} + \text{H}_2\text{O} =$
 $\text{CrCl}_3 + (\text{NH}_4)_2\text{S} + \text{H}_2\text{O} =$
5. $2\text{Cr}(\text{OH})_3 + 2\text{KNO}_3 + \text{Na}_2\text{CO}_3 = \text{K}_2\text{CrO}_4 + \text{Na}_2\text{CrO}_4 + 3\text{H}_2\text{O}$
 $+ \text{N}_2\text{O}_2 + \text{CO}_2$
 $\text{Cr}(\text{OH})_3 + \text{KNO}_3 + \text{K}_2\text{CO}_3 =$
 $\text{K}_2\text{CrO}_4 + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$

ALUMINUM, Al^{iii} = 27.1

Aluminum is a white metal which is largely used in the metallic state. Its compounds are generally colorless.

Aluminum readily forms double sulphates, called alums, with monad metals or ammonium. Similar double sulphates of other triad and monad metals are also called alums.

IMPORTANT COMPOUNDS OF ALUMINUM.

Aluminum-ammonium sulphate, alum, U. S. P., ammonium alum, $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminum chloride, N. F., AlCl_3 .

Aluminum hydroxide, U. S. P., $\text{Al}(\text{OH})_3$.

Aluminum oxide, native in corundum, emery, etc., Al_2O_3 .

Aluminum-potassium sulphate, alum, U. S. P., potassium alum, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminum silicates, native in clays, etc., composition variable.

Aluminum-sodium sulphate, sodium alum, $\text{AlNa}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminum sulphate, N. F., $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$.

Exsiccated alum, U. S. P., "burnt alum," "dried alum," $\text{AlNH}_4(\text{SO}_4)_2$ or $\text{AlK}(\text{SO}_4)_2$.

TESTS FOR ALUMINUM.

Use a separate portion of a solution of an aluminum salt, as $\text{Al}_2(\text{SO}_4)_3$, for each of the following tests:

solution and boil. A hydroxide, $\text{Al}(\text{OH})_3$ will

um solution. A white ide, $\text{Al}(\text{OH})_3$, will be of the reagent forming

ution. A white gelati- $(\text{OH})_3$, will be formed

solution. The white all be precipitated and

um solution. A white ill form. This precipi- ons of fixed alkalies, but

WING EQUATIONS:

6.

of the metals.

um, chromium, ferrous

few drops of HNO_3 T.S. iron, if present. Then excess of NH_4OH T.S. to the precipitate on the after observing that it necessary excess of this

B. Puncture the filter and transfer the precipitate to a small porcelain evaporating dish with about 5 mls of water. Add about 2 mls of NaOH T.S., boil for several minutes and filter, if necessary.

C. Acidulate the filtrate from *B* with HCl T.S., then add an excess of $(\text{NH}_4)_2\text{CO}_3$ T.S. and boil. A white precipitate shows the presence of aluminum.

D. Wash the precipitate from *B* on the filter paper and allow it to drain. Transfer a part of the moist residue to the cover of a porcelain crucible, cover with about the same amount of dry KNO_3 and about twice as much dry Na_2CO_3 , place the cover on a pipe-stem triangle supported on a tripod and heat in a gas flame until the mass is fused. Cool, then heat the cover and adhering mass with about 10 mls of water contained in a small beaker. Remove the cover from the liquid and filter.

E. Acidulate the filtrate from *D* with $\text{HC}_2\text{H}_3\text{O}_2$ T.S. and add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. A yellow precipitate shows the presence of chromium.

F. Wash any residue from *D* on the filter and allow it to drain. Pass about 5 mls of HCl T.S. through the filter and add KCNS T.S. to the filtrate. A red coloration shows the presence of iron.

G. If iron is found, test separate portions of the original solution for ferrous and ferric salts as follows:

Acidulate one portion with HCl T.S. and add $\text{K}_3\text{Fe}(\text{CN})_6$ T.S. A dark blue precipitate shows the presence of ferrous iron.

Acidulate another portion with HCl T.S. and add KCNS T.S. A red coloration shows the presence of ferric iron.

NOTE ON GROUP 4.

Some metals of the fourth and fifth groups are precipitated with the fourth group if either phosphoric acid or oxalic acid is present, as their phosphates and oxalates are insoluble in alkaline liquids; so, in the separation of all the groups, it is necessary to test the original solution for phosphoric acid and oxalic acid and remove these acids, if present, by methods to be described later, before precipitating Group 4.

PR 7.

3 and 4 of the metals.

tion to litmus. If alkaline add
action is acid. Add HCl as long

filtrate

ing as a precipitate is produced.
filter.

and filter.

filtrate
Sn, As,
h, Pt.

Examine for
of group 3
by III

Filtrate,

Boil to expel H_2S .
Add a few drops of
 HNO_3 and boil
again, then add
 NH_4Cl and an
excess of NH_4OH
and boil again.
Filter.

Precipitate,
Al, Cr, Fe.
Examine for
metals of group 4
by IV

uced by HCl in a neutral or

water, discarding the washings.
ned precipitate on the filter.

idue— $HgCl$, $AgCl$

on of NH_4OH through the residue
filter several times

+ Hg ,

Filtrate, $(NH_4)_3(AgCl)_2$
Add an excess of HNO_3 ,
white ppt., $AgCl$, shows
silver

cury

II.

Examination of any residue insoluble in $(\text{NH}_4)_2\text{S}_x$ from a precipitate produced by H_2S in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of $(\text{NH}_4)_2\text{S}_x$ and then with water, and drain, discarding the washings.

Pour a portion of hot HNO_3 through the filter several times.

Residue—HgS	Filtrate—Pb(NO₃)₂, Bi(NO₃)₃, Cu(NO₃)₂, Cd(NO₃)₂			
Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add SnCl_2 . A white or gray ppt. $\text{HgCl} + \text{Hg}$ shows	Add conc. H_2SO_4 and boil until white fumes are given off, cool, dilute with H_2O and filter.			
	Precipitate—PbSO₄	Filtrate—Bi₂(SO₄)₃, CuSO₄, CdSO₄		
	White. Dissolve with $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ and add $\text{K}_2\text{Cr}_2\text{O}_7$. Yellow ppt., PbCrO_4 , shows	Add an excess of NH_4OH and filter.		
		Precipitate, Bi(OH)₃	Filtrate—Cu(NH₃)₄SO₄, Cd(NH₃)₄SO₄	
		White. Add NaOH and SnCl_2 on filter. Brown or black coloration, Bi_2O_3 , shows	Divide into two portions.	
			To one portion add an excess of $\text{HC}_2\text{H}_3\text{O}_2$ and then $\text{K}_4\text{Fe}(\text{CN})_6$. A red ppt. or coloration, $\text{Cu}_2\text{Fe}(\text{CN})_6$, shows	Pass H_2S through the other portion, decolorizing with KCN , if Cu is present. A yellow ppt., CdS , shows
mercuric mercury	lead	bismuth	copper	cadmium

III.

Examination of the solution in $(\text{NH}_4)_2\text{S}_x$ of any precipitate produced by H_2S in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the $(\text{NH}_4)_2\text{S}_x$ solution with HCl and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in $(\text{NH}_4)_2\text{CO}_3$ T.S. Filter.

Filtrate—(NH₄)₃AsS₄	Residue—Sb₂S₃, SnS₂, Au₂S₃, PtS₂			
Add excess of HCl and pass H_2S . A yellow ppt., As_2S_3 , shows	Wash with water. Digest in concentrated HCl . Filter.			
	Filtrate—SbCl₃, SnCl₂	Residue—Au₂S₃, PtS₂		
	Place in H generator. Add H_2O and Zn . Conduct gas into AgNO_3 for three minutes	If light colored Au and Pt are absent. If dark colored, digest in nitrohydrochloric acid. Add H_2O and filter.		
	If black ppt. forms in AgNO_3 dissolve in HCl and pass H_2S . An orange ppt., Sb_2S_3 , shows	Filter liquid from generator and add few drops to HgCl_2 . A white or gray ppt., $\text{HgCl} + \text{Hg}$, shows	To a part of the filtrate add SnCl_2 and SnCl_4 and let stand. A purple ppt., Au_2O , etc., shows	
			Neutralize another part of the filtrate with NH_4OH , add alcohol and NH_4Cl . A yellow ppt., $(\text{NH}_4)_2\text{PtCl}_6$, shows	
arsenic	antimony	tin	gold	platinum

by NH_4OH , after groups from a solution.

and drain, discarding the

tes and filter.

$\text{Fe}(\text{OH})_3$

drying the washings. Transfer in crucible cover, add about twice as much dry Fe_2O_3 , heat the mass with H_2O

due— Fe_2O_3

with water, discarding the $\text{Fe}(\text{OH})_3$ in HCl and add KCNS . $\text{Fe}(\text{CNS})_3$, shows

iron

test separate portions of the solution as follows:

To another portion add HCl and KCNS . A red coloration, $\text{Fe}(\text{CNS})_3$, shows

ferric iron

ammonium sulphide, $(\text{NH}_4)_2\text{S}$,

H_4OH :

inc, Zn.

97

nickel, which is seldom constituent of the pigment. The cobaltic compounds, three, are unimportant. Cobalt has a valence of two. in color when hydrated as.

COBALT.

TESTS FOR COBALT.

Use a separate portion of a solution of a cobalt salt, as cobalt nitrate, $\text{Co}(\text{NO}_3)_2$, for each of the following tests:

1. Add $(\text{NH}_4)_2\text{S}$ T.S. to the cobalt solution. A black precipitate of cobalt sulphide, CoS , will form, which is insoluble in an excess of the reagent and in diluted HCl , but is soluble in concentrated HCl and in diluted HNO_3 .

2. Add NaOH T.S. to the cobalt solution and boil the liquid. A blue precipitate of a basic salt is obtained which forms cobalt hydroxide, $\text{Co}(\text{OH})_2$, and becomes red in color upon boiling.

3. Slowly add NH_4OH T.S. to the cobalt solution. A precipitate of cobalt hydroxide, $\text{Co}(\text{OH})_2$, is produced, but it immediately dissolves in an excess of the reagent to form a brown solution containing complex double compounds of variable composition.

4. Acidulate a portion of the cobalt solution with acetic acid, and add twice its volume of KNO_2 T.S. Warm the mixture and allow it to stand for some time. A yellow crystalline precipitate of potassium cobaltic nitrite, $\text{K}_3\text{Co}(\text{NO}_2)_6$, will slowly form. This compound is soluble in water but is insoluble in an excess of KNO_2 .

5. Bend the end of a piece of platinum wire around the point of a lead pencil to form a loop about 3 millimeters in diameter. Heat this loop in the oxidizing blowpipe flame for a moment and plunge it into some powdered borax. Heat the adhering borax in the blowpipe flame until it fuses to form a clear and colorless glass-like bead. Dip this bead in the cobalt solution and again heat it in the blowpipe flame. The bead will be colored dark blue. If too much cobalt is present the color will appear to be black.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Co}(\text{NO}_3)_2 + (\text{NH}_4)_2\text{S} =$
 $\text{CoSO}_4 + (\text{NH}_4)_2\text{S} =$
2. $\text{Co}(\text{NO}_3)_2 + \text{NaOH} =$
 $\text{CoCl}_2 + \text{KOH} =$
3. $\text{CoCl}_2 + \text{NH}_4\text{OH} =$
 $\text{CoSO}_4 + \text{NH}_4\text{OH} =$
4. $\text{CoCl}_2 + 7\text{KNO}_2 + 2\text{HC}_2\text{H}_3\text{O}_2 = \text{K}_3\text{Co}(\text{NO}_2)_6 + 2\text{KCl} +$
 $2\text{KC}_2\text{H}_3\text{O}_2 + \text{H}_2\text{O} + \text{NO}$

NICKEL, $\text{Ni}^{\text{ii, iii}}$ = 58.68

Nickel is a silvery white metal which is largely used in the metallic state for electroplating and as a constituent of various alloys. German silver is an alloy of copper, zinc and nickel. United States "nickel" coins are 75 per cent. copper and 25 per cent. nickel.

...ands, only those in which it
...ompounds of nickel gener-
...impart to their solutions.

... OF NICKEL.

...
...₄)₂.

... EL.

...a nickel salt, as nickel chlo-
...:

...tion. A black precipitate
...This precipitate is insol-
...ehloric acid, but is readily
...acid.

...on. A pale green precipi-
...formed, which is insoluble

...nickel solution. The pale
...OH)₂, will be formed which
...blue solution in an excess

...nickel solution and heat it in
...bead will be colored violet
....

... FLOWING EQUATIONS:



vi, vii = 54.93

...embling cast iron, which is
...constituent of some varie-
...in valence, forming several
...important are the manganous

compounds in which it has a valence of two, and the permanganates in which it has a valence of seven.

The manganous compounds generally have a light pink color. Other compounds of manganese have various bright colors.

IMPORTANT COMPOUNDS OF MANGANESE.

Manganese chloride, manganous chloride, $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$.

Manganese dioxide, precipitated manganese dioxide, U. S. P., pyrolusite, MnO_2 .

Manganese hypophosphite, N.F., manganous hypophosphite, $\text{Mn}(\text{PH}_2\text{O}_2)_2 \cdot \text{H}_2\text{O}$.

Manganese sulphate, N. F., manganous sulphate, $\text{MnSO}_4 \cdot \text{H}_2\text{O}$.

Potassium manganate, K_2MnO_4 .

Potassium permanganate, KMnO_4 .

TESTS FOR MANGANOUS SALTS.

Use a separate portion of a solution of a manganous salt, as MnCl_2 , for each of tests 1, 2, 3 and 4:

1. Add $(\text{NH}_4)_2\text{S}$ T.S. to the manganese solution. A flesh-colored precipitate of manganous sulphide, MnS , will be formed. Manganous sulphide is readily soluble in diluted mineral acids and in acetic acid.

2. Add NaOH T.S. to the manganese solution. A white precipitate of manganous hydroxide, $\text{Mn}(\text{OH})_2$, will be formed; which oxidizes to form basic manganic hydroxide, $\text{MnO}(\text{OH})$, and turns brown upon exposure to air. Both hydroxides are insoluble in an excess of the reagent.

Collect some of the precipitated $\text{Mn}(\text{OH})_2$ on a filter paper for use in tests 5 and 6.

3. Add NH_4OH T.S. to the manganese solution. A part of the manganese is precipitated as the white manganous hydroxide, $\text{Mn}(\text{OH})_2$, which oxidizes and turns brown upon exposure to air. The other part forms a double salt and remains in solution.

To a portion of the manganese solution add double its volume of NH_4Cl T.S. and then add NH_4OH T.S. The manganese forms a soluble double salt, manganous ammonium chloride, $\text{Mn}(\text{NH}_4)_2\text{Cl}_4$, and precipitation does not occur until oxidation takes place when the brown basic manganic hydroxide, $\text{MnO}(\text{OH})$, is slowly precipitated.

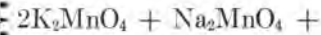
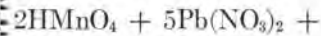
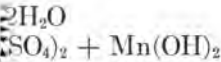
4. Add Na_2CO_3 T.S. to the manganese solution. A white precipitate of manganous carbonate, MnCO_3 , will form, which oxidizes to form the brown basic manganic hydroxide, $\text{MnO}(\text{OH})$, upon exposure to air.

5. Mix about one-quarter of a teaspoonful of lead peroxide, PbO_2 , with about 5 mls of HNO_3 T.S. and a small quantity of the pre-

and boil. Allow the powder
ant liquid has the pink or
 MnO_4 . The $\text{Mn}(\text{OH})_2$ dis-
oxidized to HMnO_4 .

he precipitated $\text{Mn}(\text{OH})_2$
crucible and cover it with
twice as much dry Na_2CO_3 .
le and heat in the flame of
cool. The manganese is
assium nitrate and a green
anganates will be obtained.

FOLLOWING EQUATIONS:

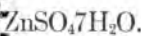


37

which is largely used in the
per and zinc. Galvanized
salts are poisonous and are

OS OF ZINC.

bonate, U. S. P., basic zinc



TESTS FOR ZINC.

Use a separate portion of a solution of a zinc salt, as ZnSO_4 , for each of tests 1, 2, 3 and 4.

1. Add $(\text{NH}_4)_2\text{S}$ T.S. to the zinc solution. A white precipitate of zinc sulphide, ZnS , will be obtained. The precipitate is soluble in mineral acids, but is not soluble in acetic acid.

2. Slowly add NaOH T.S. to the zinc solution. A white precipitate of zinc hydroxide, $\text{Zn}(\text{OH})_2$ is obtained, which forms sodium zincate, Na_2ZnO_2 , and dissolves in an excess of the reagent. The zinc is partially re-precipitated as zinc oxide, ZnO , upon boiling the solution.

3. Slowly add NH_4OH T.S. to the zinc solution until the precipitate formed is re-dissolved, then boil the solution obtained. The first precipitate formed is zinc hydroxide, $\text{Zn}(\text{OH})_2$. This dissolves in an excess of NH_4OH to form ammonium zincate, $(\text{NH}_4)_2\text{ZnO}_2$. Finally a precipitate of zinc oxide, ZnO , is formed upon boiling the solution.

4. Add Na_2CO_3 T.S. to the zinc solution. A white precipitate of zinc sub-carbonate, $(\text{ZnCO}_3)_2(\text{Zn}(\text{OH})_2)_3$, is obtained. This precipitate is soluble in acids and in alkali hydroxides.

Collect some of the precipitated zinc sub-carbonate on a small filter paper, wash with water and allow to drain for use in test 5.

5. Fold the filter paper containing the precipitate collected from test 4, place it on the cover of a porcelain crucible with a small amount of dry Na_2CO_3 and add a few drops of $\text{Co}(\text{NO}_3)_2$ T.S. Place the cover on a pipe-stem triangle supported on a tripod and ignite in the flame of a gas burner. A green mass consisting of zinc and cobalt oxides will be obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{ZnSO}_4 + (\text{NH}_4)_2\text{S} =$
 $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{K}_2\text{S} =$
2. $\text{ZnCl}_2 + \text{NaOH} =$
 $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{KOH} =$
 $\text{Zn}(\text{OH})_2 + \text{NaOH} =$
3. $\text{ZnSO}_4 + \text{NH}_4\text{OH} =$
 $\text{Zn}(\text{OH})_2 + \text{NH}_4\text{OH} =$
 $(\text{NH}_4)_2\text{ZnO}_2 + \text{heat} =$
4. $5\text{ZnSO}_4 + 5\text{Na}_2\text{CO}_3 + 3\text{H}_2\text{O} = (\text{ZnCO}_3)_2(\text{Zn}(\text{OH})_2)_3 + 5\text{Na}_2\text{SO}_4$
 $+ 3\text{CO}_2$
 $\text{ZnCl}_2 + \text{K}_2\text{CO}_3 =$

of the metals.

the following metals:

(NH₄)₂S T.S. If no are absent. If a pre-
gent until precipitation
filtrate. If precipita-
ted.

in cold water, discarding
if about 5 mils of HCl
several times. Cobalt
dissolved, and zinc and
solve.

s to smell of H₂S. Add
ecessary. Manganese, if
zinc, if present, forms

U. If no precipitate is
precipitate, ZnS, shows

, manganese is absent.

C, transfer some of it
amount of powdered
HNO₃ T.S., and boil.
stant liquid has a pink
is shown.

the filter paper from B,
a dark-colored residue
er and wash the residue
hydrochloric acid. Add
precipitate produced on a
filtrate and washings.
H₂O₂ T.S. through the

add about 10 mils each
alf an hour and filter if
balt is absent. If KCl
Co(NO₂)₆, the presence

cess of NaOH. If no
light green precipitate

SAMPLE NUMBER 9.

Analysis of a solution for groups 1, 2, 3, 4 and 5 of the metals.

The solution should be neutral or acid in reaction to litmus. If alkaline add HNO_3 to the portion to be examined for metals until the reaction is acid. Add HCl as long as a precipitate is produced and filter.

Precipitate Pb, Hg', Ag. Examine for metals of group 1 by I.	Filtrate			
	Warm and pass H_2S as long as a precipitate is produced. Filter.			
	Precipitate		Filtrate	
	Digest with $(\text{NH}_4)_2\text{S}_x$ and filter.		Boil to expel H_2S . Add a few drops of HNO_3 and boil again; then add NH_4Cl and an excess of NH_4OH and boil again. Filter.	
	Residue Hg', Pb, Bi, Cu, Cd.	Filtrate Sb, Sn, As, Au, Pt.	Precipitate Al, Cr, Fe.	Filtrate
	Examine for metals of group 2 by II.	Examine for metals of group 3 by III	Examine for metals of group 4 by IV.	Pass H_2S as long as a ppt. is produced. Filter.
				Precipitate Zn, Mn, Co, Ni.
				Examine for metals of group 5 by V.

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.		
Filtrate—PbCl_2.	Residue—HgCl, AgCl	
Add H_2SO_4 and cool. White ppt., PbSO_4 , shows	Pass the same portion of NH_4OH through the residue on the filter several times	
	Residue—$\text{NH}_4\text{HgCl} + \text{Hg}$, black, shows	Filtrate, $(\text{NH}_4)_2(\text{AgCl})_2$. Add an excess of HNO_3 , white ppt., AgCl , shows
lead	mercurous mercury	silver

$(\text{NH}_4)_2\text{S}_x$ from a precipi-
group 1 of the metals

al portion of $(\text{NH}_4)_2\text{S}_x$ and
several times.

$\text{Cu}(\text{NO}_3)_2$, $\text{Cd}(\text{NO}_3)_2$

fumes are given off, cool,
filter.

$(\text{O}_4)_2$, CuSO_4 , CdSO_4

of NH_4OH and filter.

Filtrate—

$(\text{NH}_4)_2\text{SO}_4$, $\text{Cd}(\text{NH}_4)_2\text{SO}_4$.

Divide into two portions.

one portion
an excess

$\text{H}_2\text{C}_2\text{H}_3\text{O}_2$

and then

$\text{Fe}(\text{CN})_6$.

ed ppt. or

coloration,

$\text{Fe}(\text{CN})_6$,

shows

copper

Pass H_2S
through the
other portion,
decolorizing
with KCN , if
 Cu is present.
A yellow ppt.,
 CdS , shows

cadmium

of any precipitate pro-
of the metals has been

collect any precipitate formed
and digest in $(\text{NH}_4)_2\text{CO}_3$ T.S.

, Au_2S_3 , PtS_2

concentrated HCl . Filter.

Residue— Au_2S_3 , PtS_2

light colored Au and Pt
absent. If dark colored, di-
in nitrohydrochloric acid.
 H_2O and filter.

a part of
filtrate add
 H_2Cl_2 and
and let
and. A pur-
ppt., Au_2O_3 ,
c., shows

Neutralize
another part of
the filtrate with
 NH_4OH , add
alcohol and
 NH_4Cl . A yel-
low ppt.,
 $(\text{NH}_4)_2\text{PtCl}_6$,
shows

gold

platinum

a neutral or alkaline
 O_2 in the presence of

quickly oxidizes in the
 but many of its com-
 pounds of calcium were
 calcium phosphate was

CALCIUM.

carbonate, U. S. P., pre-
 estone, whiting, CaCO_3 .

CaO .
 phosphate, N.F., $\text{Ca}_3(\text{PO}_4)_2$.

is, CaSO_4 .

der, "chloride of lime,"

calcium salt, as CaCl_2 , for

solution. A white preci-
 e obtained, which is not
 alis, but is readily soluble
 the same precipitate.

on. A white precipitate
 ained if the solutions are
 is obtained from dilute
 water. It is less soluble

in hot water than it is in cold water. Ammonium hydroxide does not precipitate calcium from solutions of calcium salts.

3. Add H_2SO_4 T.S. to the calcium solution. A white precipitate of calcium sulphate, CaSO_4 , will be obtained, unless the solution is very dilute. CaSO_4 is slightly soluble in water. Soluble sulphates, as Na_2SO_4 , will give the same precipitate.

4. Add Na_2HPO_4 T.S. to the calcium solution. A white precipitate of calcium hydrogen phosphate, CaHPO_4 , will be obtained. The precipitate is readily soluble in acids.

5. Add $(\text{NH}_4)_2\text{C}_2\text{O}_4$ T.S. to the calcium solution. A white precipitate of calcium oxalate, CaC_2O_4 , will be obtained. The precipitate is readily soluble in mineral acids or in oxalic acid, but is not soluble in acetic acid.

6. Clean a piece of platinum wire by holding it in a blue gas flame until it ceases to give any color to the flame. Dip the wire in the calcium solution and heat again. The flame will be colored yellowish-red.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{CaCl}_2 + (\text{NH}_4)_2\text{CO}_3 =$
 $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{Na}_2\text{CO}_3 =$
2. $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{NaOH} =$
 $\text{CaBr}_2 + \text{KOH} =$
3. $\text{CaBr}_2 + \text{K}_2\text{SO}_4 =$
 $\text{CaCl}_2 + (\text{NH}_4)_2\text{SO}_4 =$
4. $\text{CaCl}_2 + \text{Na}_2\text{HPO}_4 =$
 $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{Na}_2\text{HPO}_4 =$
5. $\text{CaCl}_2 + (\text{NH}_4)_2\text{C}_2\text{O}_4 =$
 $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{K}_2\text{C}_2\text{O}_4 =$

STRONTIUM, $\text{Sr}^{\text{II}} = 87.63$

Strontium is a yellow metal which readily oxidizes in moist air and actively decomposes water. It is seldom seen in the metallic state.

IMPORTANT COMPOUNDS OF STRONTIUM.

Strontium bromide, U. S. P., $\text{SrBr}_2 \cdot 6\text{H}_2\text{O}$.

Strontium carbonate, SrCO_3 .

Strontium chloride, $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$.

Strontium iodide, U. S. P., $\text{SrI}_2 \cdot 6\text{H}_2\text{O}$.

Strontium nitrate, $\text{Sr}(\text{NO}_3)_2$.

TESTS FOR STRONTIUM.

Use separate portions of a solution of a strontium salt, as SrCl_2 , for each of the following tests:

tium solution. A white SrCO_3 , will form, which is insoluble in alkalis, but is readily

soluble in acids. A white precipitate forms if the solution is not too concentrated or alkalies.

In a solution and warm the reagent sulphate, SrSO_4 , will form. It is soluble in water, the reagent sulphate is small. As this reagent can be used to detect stron-

tium solution. A white precipitate of SrHPO_4 , will form, which

is soluble in acids. A white precipitate will form. The precipitate is very slightly soluble in

acid. It burns in luminous gas flame until it is cooled. The cleaned wire in the flame. The flame will be

WORKING EQUATIONS:

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is not used in the metallic

BARIUM.

Barium hydroxide, $\text{Ba}(\text{OH})_2$.

Barium nitrate, $\text{Ba}(\text{NO}_3)_2$.

Barium sulphate, permanent white, BaSO_4 .

Barium sulphide, BaS .

TESTS FOR BARIUM.

Use a separate portion of a solution of a barium compound, as BaCl_2 , for each of the following tests:

1. Add $(\text{NH}_4)_2\text{CO}_3$ T.S. to the barium solution. A white precipitate of barium carbonate, BaCO_3 , will be obtained. Other soluble carbonates, as Na_2CO_3 , will give the same precipitate.

2. Add H_2SO_4 T.S. to the barium solution. A white precipitate of barium sulphate, BaSO_4 , will be obtained, which is insoluble in acids or alkalis. Soluble sulphates, as K_2SO_4 , give the same precipitate.

3. Add $(\text{NH}_4)_2\text{C}_2\text{O}_4$ T.S. to the barium solution. A white precipitate of barium oxalate, BaC_2O_4 , will be obtained. The precipitate is soluble in acetic acid or in a solution of oxalic acid.

4. Add Na_2HPO_4 T.S. to the barium solution. A white precipitate of barium hydrogen phosphate, BaHPO_4 , will be obtained.

5. Add $\text{K}_2\text{Cr}_2\text{O}_7$ T.S. to the barium solution. A yellow precipitate of barium chromate, BaCrO_4 , will be obtained. This precipitate is soluble in nitric and hydrochloric acids, but is insoluble in acetic acid. K_2CrO_4 T.S. will give the same precipitate.

6. Make a flame test of the barium solution with platinum wire. The flame will be colored a yellowish-green.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{BaCl}_2 + (\text{NH}_4)_2\text{CO}_3 =$
 $\text{Ba}(\text{NO}_3)_2 + \text{Na}_2\text{CO}_3 =$
2. $\text{Ba}(\text{NO}_3)_2 + \text{H}_2\text{SO}_4 =$
 $\text{BaCl}_2 + \text{K}_2\text{SO}_4 =$
3. $\text{Ba}(\text{C}_2\text{H}_3\text{O}_2)_2 + (\text{NH}_4)_2\text{C}_2\text{O}_4 =$
 $\text{BaCl}_2 + \text{K}_2\text{C}_2\text{O}_4 =$
4. $\text{BaCl}_2 + \text{Na}_2\text{HPO}_4 =$
 $\text{Ba}(\text{NO}_3)_2 + (\text{NH}_4)_2\text{HPO}_4 =$
5. $\text{BaCl}_2 + \text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{O} =$
 $\text{Ba}(\text{NO}_3)_2 + \text{K}_2\text{CrO}_4 =$

SAMPLE NUMBER 10.

Analysis of a solution for group 6 of the metals.

The solution may contain salts of any of the following metals: calcium, strontium, barium.

A. To about 10 mls of the sample add about 5 mls of NH_4Cl T.S. and about 5 mls of NH_4OH T.S. and then $(\text{NH}_4)_2\text{CO}_3$ T.S. If no

and barium are absent. Add $(\text{NH}_4)_2\text{CO}_3$ T.S. until the solution is alkaline and test the filtrate. Add water on the filter with water, a portion of about 5 mls of water to dissolve the precipitate to the solution. If no precipitate is present. A yellow precipitate is present continue to add until the reaction is complete. Filter the filtrate into two portions. From *B* add CaSO_4 T.S., if a precipitate is produced, strontium is present. From *B*, add K_2SO_4 T.S., if a precipitate is produced, to remove strontium if present. T.S. If no precipitate is produced, amount of white precipitate is small.

11.

4, 5 and 6 of the metals.

ion to litmus. If alkaline add water until the reaction is acid. Add

precipitate is produced. Filter.

Filtrate

to expel H_2S . Add a few drops of O_2 and boil again, then add an excess of NH_4OH and filter.

Filtrate

Pass H_2S as long as a ppt. is produced. Filter.

Precipitate
Zn, Mn,
Co, Ni.

Examine
for metals
of group 5
by V.

Filtrate

Boil to expel
 H_2S , add an
excess of
 $(\text{NH}_4)_2\text{CO}_3$
and filter.

Precipitate
Ba, Sr, Ca.

Examine
for metals
of group 6
by VI.

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.		
Filtrate—PbCl₂	Residue—HgCl, AgCl	
Add H ₂ SO ₄ and cool. White ppt. PbSO ₄ , shows	Pass the same portion of NH ₄ OH through the residue on the filter several times	
	Residue—NH₂HgCl+Hg, black, shows	Filtrate, (NH₄)₂(AgCl)₂ Add an excess of HNO ₃ , white ppt., AgCl, shows
lead	mercurous mercury	silver

II.

Examination of any residue insoluble in (NH₄)₂S_x from a precipitate produced by H₂S in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of (NH₄)₂S_x and then with water, and drain, discarding the washings.
Pour a portion of hot HNO₃ through the filter several times.

Residue, HgS	Filtrate—Pb(NO₃)₂, Bi(NO₃)₃, Cu(NO₃)₂, Cd(NO₃)₂			
Black. Dissolve in nitrohydrochloric acid, boil to expel Cl, dilute with water, filter and add SnCl ₂ . A white or gray ppt. HgCl+Hg shows	Add conc. H ₂ SO ₄ and boil until white fumes are given off, dilute with H ₂ O, cool and filter.			
	Precipitate, PbSO₄	Filtrate—Bi₂(SO₄)₃, CuSO₄, CdSO₄		
	White. Dissolve with NH ₄ C ₂ H ₃ O ₂ and add K ₂ Cr ₂ O ₇ . Yellow ppt., PbCrO ₄ , shows	Add an excess of NH ₄ OH and filter.		
		Precipitate, Bi(OH)₃	Filtrate—	
		White. Add NaOH and SnCl ₂ on filter. Brown or black coloration, Bi ₂ O ₃ , shows	Cu(NH₄)₂SO₄, Cd(NH₄)₂SO₄	
			Divide into two portions.	
			To one portion add an excess of HC ₂ H ₃ O ₂ and then K ₄ Fe(CN) ₆ . A red ppt. or coloration Cu ₂ Fe(CN) ₆ , shows	Pass H ₂ S through the other portion, first decolorizing with KCN if Cu is present. A yellow ppt., CdS, shows
mercuric mercury	lead	bismuth	copper	cadmium

III.

Examination of the solution in $(\text{NH}_4)_2\text{S}_x$ of any precipitate produced by H_2S in an acid liquid, after group 1 of the metals has been removed from a solution.

Acidulate the $(\text{NH}_4)_2\text{S}_x$ solution with HCl and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in $(\text{NH}_4)_2\text{CO}_3$ T.S. Filter.

Filtrate— $(\text{NH}_4)_2\text{AsS}_4$		Residue— Sb_2S_3 , SnS_2 , Au_2S_3 , PtS_2		
Add excess of HCl and pass H_2S . A yellow ppt., As_2S_3 , shows		Wash with water. Digest in concentrated HCl . Filter.		
		Filtrate— SbCl_3 , SnCl_2	Residue— Au_2S_3 , PtS_2	
		Place in H generator. Add H_2O and Zn . Conduct gas into AgNO_3 for three minutes	If light colored Au and Pt are absent. If dark colored, digest in nitrohydrochloric acid. Add H_2O and filter.	
		If black ppt. forms in AgNO_3 dissolve in HCl and pass H_2S . An orange ppt., Sb_2S_3 , shows	Filter liquid from generator and add few drops to HgCl_2 . A white or gray ppt., $\text{HgCl}_2 + \text{Hg}$, shows	To a part of the filtrate add SnCl_2 and SnCl_4 and let stand. A purple ppt., Au_2O_3 , etc., shows
				Neutralize another part of the filtrate with NH_4OH , add alcohol and NH_4Cl . A yellow ppt., $(\text{NH}_4)_2\text{PtCl}_6$, shows
arsenic	antimony	tin	gold	platinum

IV.

Examination of any precipitate produced by NH_4OH , after groups 1, 2 and 3 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Boil the precipitate with NaOH for several minutes and filter.

Filtrate, NaAlO_2 .		Residue— $\text{Cr}(\text{OH})_3$, $\text{Fe}(\text{OH})_3$	
Acidulate with HCl , then add an excess of $(\text{NH}_4)_2\text{CO}_3$ and boil. A white ppt., $\text{Al}(\text{OH})_3$, shows		Wash with H_2O and drain, discarding the washings. Transfer a part of the residue to a porcelain crucible cover, add about the same amount of dry KNO_3 and about twice as much dry Na_2CO_3 and ignite until fused. Cool, heat the mass with H_2O and filter.	
		Filtrate, K_2CrO_4 .	Residue— Fe_2O_3
		Acidulate with HCl and add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$. A yellow ppt., PbCrO_4 , shows	Wash on the filter with water, discarding the washings. Dissolve in HCl and add KCNS . A red coloration, $\text{Fe}(\text{CNS})_3$, shows
			iron
			If iron is present test separate portions of the original solution as follows:
		To one portion add HCl and $\text{K}_3\text{Fe}(\text{CN})_6$. A dark blue ppt., $\text{Fe}_3(\text{Fe}(\text{CN})_6)_2$, shows	To another portion add HCl and KCNS . A red coloration, $\text{Fe}(\text{CNS})_3$, shows
aluminum	chromium	ferrous iron	ferric iron

V.

Examination of any precipitate produced by H_2S in an alkaline liquid, after groups 1, 2, 3 and 4 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Pass the same portion of HCl through the filter several times.

Filtrate— $ZnCl_2$, $MnCl_2$.		Residue— CoS , NiS .	
Boil to expel H_2S , cool, add excess of $NaOH$ and filter.		If light colored, cobalt and nickel are absent. If dark colored, dissolve in nitro-hydrochloric acid, add an excess of $NaOH$, collect the ppt. produced on a filter and wash, discarding the washings. Dissolve the ppt. in $HC_2H_3O_2$, add an excess of KCl and of KNO_3 , let stand for half an hour and filter.	
Filtrate, Na_2ZnO_2.	Residue, $Mn(OH)_2$.	Precipitate, $K_2Co(NO_2)_6$ yellow, shows	Filtrate, $Ni(C_2H_3O_2)_2$ Add an excess of $NaOH$. Light green ppt. $Ni(OH)_2$, shows
Pass H_2S . A white ppt. ZnS , shows	Boil with conc. HNO_3 and PbO_2 and let stand. A pink supernatant liquid shows		
zinc	manganese	cobalt	nickel

VI.

Examination of any precipitate produced by $(NH_4)_2CO_3$, after groups 1, 2, 3, 4 and 5 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings.

Dissolve the washed precipitate in $HC_2H_3O_2$, add an excess of $K_2Cr_2O_7$ and filter.

Precipitate, $BaCrO_4$	Filtrate—$Sr(C_2H_3O_2)_2$, $Ca(C_2H_3O_2)_2$.	
Yellow shows	Divide into two portions.	
barium	To one portion add $CaSO_4$, boil and let stand. White ppt. $SrSO_4$, shows	To the other portion, add K_2SO_4 , boil, cool and filter, if necessary. To the filtrate add $(NH_4)_2C_2O_4$. White ppt. CaC_2O_4 , shows
	strontium	calcium

GROUP 7.

Metals not precipitated by any group reagent:

Magnesium, Mg ; potassium, K ; sodium, Na ; lithium, Li ; ammonium, $(NH_4)^+$.

MAGNESIUM, $Mg^{ii} = 24.32$

Magnesium is a silvery-white metal of low specific gravity which is permanent in dry air but oxidizes in moist air. It burns in the air,

giving an intense white light. It is a constituent of flashlight powders, but is most important in its compounds.

IMPORTANT COMPOUNDS OF MAGNESIUM.

Magnesium carbonate, native as magnesite, MgCO_3 .

Magnesium sub-carbonate, magnesium carbonate, U. S. P., *magnesia alba*, $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$.

Magnesium chloride, N.F., $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$.

Magnesium hydroxide, $\text{Mg}(\text{OH})_2$.

Magnesium oxide, U. S. P., *magnesia*, calcined *magnesia*, MgO .

Heavy magnesium oxide, U. S. P., heavy *magnesia*, MgO .

Magnesium sulphate, U. S. P., *epsom salt*, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

Magnesium silicates, talcum, asbestos, *meerschaum*, soapstone; variable in composition.

TESTS FOR MAGNESIUM.

Use a separate portion of a solution of a salt of magnesium, as MgCl_2 , for each of the following tests:

1. Add $(\text{NH}_4)_2\text{CO}_3$ T.S. to the magnesium solution. A white precipitate of magnesium-ammonium carbonate, $\text{MgCO}_3(\text{NH}_4)_2\text{CO}_3$, is formed when the reagent is in excess. No precipitate will form if excessive amounts of ammonium salts are present.

To a portion of the magnesium solution add an equal volume of NH_4Cl T.S. and then $(\text{NH}_4)_2\text{CO}_3$ T.S. Soluble magnesium-ammonium chloride is formed and this is not precipitated by the carbonate.

2. Add Na_2CO_3 T.S. to the magnesium solution. A white precipitate of magnesium sub-carbonate, principally $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2$ is formed. The precipitate is soluble in solutions of ammonium salts and will not form if they are present.

3. Add NH_4OH T.S. to the magnesium solution. Half of the magnesium is precipitated as the white gelatinous magnesium hydroxide, $\text{Mg}(\text{OH})_2$. The other half forms a double salt, as $(\text{NH}_4)_2\text{MgCl}_4$, and remains in solution. The precipitate forms soluble double compounds with ammonium salts and no precipitation occurs if an excess of ammonium salts is present in the solution.

4. Add NaOH T.S. to the magnesium solution. A gelatinous white precipitate of magnesium hydroxide, $\text{Mg}(\text{OH})_2$, will form. The precipitate is soluble in ammonium salts and will not form if they are present.

5. To a portion of the magnesium solution add NH_4Cl T.S., NH_4OH T.S., and Na_2HPO_4 T.S. in the order given. A white crystalline precipitate of magnesium-ammonium phosphate, MgNH_4PO_4 , will form slowly. NH_4Cl does not take part in the reaction, but is used to prevent precipitation of magnesium hydroxide by NH_4OH .

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{MgCl}_2 + (\text{NH}_4)_2\text{CO}_3 =$
 $\text{MgSO}_4 + (\text{NH}_4)_2\text{CO}_3 =$
 $\text{MgCl}_2 + \text{NH}_4\text{Cl} =$
2. $\text{MgCl}_2 + \text{K}_2\text{CO}_3 =$
 $\text{MgSO}_4 + \text{Na}_2\text{CO}_3 =$
3. $\text{MgBr}_2 + \text{NH}_4\text{OH} =$
 $\text{MgSO}_4 + \text{NH}_4\text{OH} =$
4. $\text{MgSO}_4 + \text{NaOH} =$
 $\text{MgCl}_2 + \text{KOH} =$
5. $\text{MgCl}_2 + \text{NH}_4\text{OH} + \text{Na}_2\text{HPO}_4 =$
 $\text{MgSO}_4 + \text{NH}_4\text{OH} + (\text{NH}_4)_2\text{HPO}_4 =$

POTASSIUM (KALIUM), $K^i = 39.10$

Potassium is the most electropositive of the metals which are found in any considerable quantity. It is a soft white metal which must be kept under kerosene, as it rapidly oxidizes and takes fire in contact with water or when exposed to the air. The element is but little used in the metallic state. The salts of potassium are very important and all of them are more or less soluble in water.

IMPORTANT COMPOUNDS OF POTASSIUM.

Potassium acetate, U. S. P., $\text{KC}_2\text{H}_3\text{O}_2$.
 Potassium bicarbonate, U. S. P., saleratus, KHCO_3 .
 Potassium bitartrate, U. S. P., cream of tartar, $\text{KHC}_4\text{H}_4\text{O}_6$.
 Potassium bromate, KBrO_3 .
 Potassium bromide, U. S. P., KBr .
 Potassium carbonate, U. S. P., potash, pearlash, salt of tartar, K_2CO_3 .
 Potassium chlorate, U. S. P., KClO_3 .
 Potassium chloride, N.F., KCl .
 Potassium chromate, K_2CrO_4 .
 Potassium cyanide, prussiate of potash, KCN .
 Potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$.
 Potassium ferricyanide, red prussiate of potash, $\text{K}_3\text{Fe}(\text{CN})_6$.
 Potassium ferrocyanide, yellow prussiate of potash, $\text{K}_4\text{Fe}(\text{CN})_6$.
 Potassium hydroxide, U. S. P., potassium hydrate, potassa, caustic potash, KOH .
 Potassium hypophosphite, U. S. P., KPH_2O_2 .
 Potassium iodide, U. S. P., KI .
 Potassium nitrate, U. S. P., saltpetre, nitre, KNO_3 .
 Potassium perchlorate, KClO_4 .
 Potassium permanganate, U. S. P., KMnO_4 .
 Potassium sulphate, N.F., K_2SO_4 .

Potassium sulphocyanate, potassium thiocyanate, KCNS.

Potassium and sodium tartrate, U. S. P., rochelle salt, $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$.

Sulphuretted potash, U.S.P., liver of sulphur, composition indefinite.

TESTS FOR POTASSIUM.

Use a separate portion of a solution of a potassium salt, as KCl for each of the following tests:

1. Add $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ T.S. to the potassium solution. A white precipitate of potassium bi-tartrate, $\text{KHC}_4\text{H}_4\text{O}_6$, will form if the solution is not too dilute. The addition of an equal volume of alcohol increases the delicacy of the test, as the precipitate is less soluble in diluted alcohol than it is in water. The precipitate is soluble in alkalis, and will not form if they are present in the solution until an excess of the reagent has been added. Sodium hydrogen tartrate, $\text{NaHC}_4\text{H}_4\text{O}_6$, will produce the same precipitate in neutral or acid solutions.

2. Acidulate a portion of the potassium solution with $\text{HC}_2\text{H}_3\text{O}_2$ T.S., add $\text{Na}_3\text{Co}(\text{NO}_2)_6$ T.S. and let stand. A yellow precipitate of potassium-cobaltic nitrite, $\text{K}_3\text{Co}(\text{NO}_2)_6$, will form slowly, if the solution is not too dilute.

3. Heat some of the potassium solution on platinum wire in a blue gas flame and observe the color of the flame, directly and through blue glass. A characteristic violet flame, visible through blue glass, is obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{KCl} + \text{H}_2\text{C}_4\text{H}_4\text{O}_6 =$
 $\text{K}_2\text{SO}_4 + \text{NaHC}_4\text{H}_4\text{O}_6 =$
2. $\text{KC}_2\text{H}_3\text{O}_2 + \text{Na}_3\text{Co}(\text{NO}_2)_6 =$
 $\text{KBr} + \text{Na}_3\text{Co}(\text{NO}_2)_6 =$

SODIUM (NATRIUM), $\text{Na}^1 = 23.0$

Sodium is a soft white metal, closely resembling potassium. It decomposes water and rapidly oxidizes in the air, so it must be kept under kerosene or some other liquid which does not contain oxygen. The compounds of sodium are very important and all of them are soluble in water.

IMPORTANT COMPOUNDS OF SODIUM.

Sodium acetate, U. S. P., $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$.

Sodium arsenate, U. S. P., $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$.

Exsiccated sodium arsenate, U. S. P., Na_2HAsO_4 .

Sodium bicarbonate, U. S. P., baking soda, NaHCO_3 .
 Sodium bisulphite, NaHSO_3 .
 Sodium borate, U. S. P., sodium tetraborate, sodium pyroborate, borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$.
 Sodium bromide, U. S. P., NaBr .
 Sodium carbonate, sal soda, washing soda, $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.
 Monohydrated sodium carbonate, U. S. P., $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$.
 Sodium chlorate, NaClO_3 .
 Sodium chloride, U. S. P., salt, NaCl .
 Sodium cyanide, U. S. P., NaCN .
 Sodium hydroxide, U. S. P., sodium hydrate, caustic soda, soda, NaOH .
 Sodium hypochlorite, NaOCl .
 Sodium hypophosphite, U. S. P., $\text{NaPH}_2\text{O}_2 \cdot \text{H}_2\text{O}$.
 Sodium iodide, U. S. P., NaI .
 Sodium nitrate, U. S. P., Chili saltpetre, NaNO_3 .
 Sodium nitrite, U. S. P., NaNO_2 .
 Sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$.
 Sodium perborate, U. S. P., $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$.
 Sodium peroxide, Na_2O_2 .
 Tri-sodium phosphate, Na_3PO_4 .
 Sodium phosphate, U. S. P., di-sodium hydrogen phosphate, $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$.
 Exsiccated sodium phosphate, U. S. P., Na_2HPO_4 .
 Sodium di-hydrogen phosphate, NaH_2PO_4 .
 Sodium pyrophosphate, $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$.
 Sodium silicates, etc., water glass, soluble glass, Na_2SiO_3 , $\text{Na}_2\text{Si}_4\text{O}_9$.
 Sodium sulphate, U. S. P., Glauber's salt, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$.
 Sodium sulphide, $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$.
 Sodium sulphite, $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$.
 Exsiccated sodium sulphite, U. S. P., Na_2SO_3 .
 Sodium tartrate, $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$.
 Sodium thiosulphate, U. S. P., "hyposulphite of soda," $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

TEST FOR SODIUM.

As all compounds of sodium are soluble in water to an appreciable extent, there are no dependable precipitation tests for the metal. The flame test is very sensitive, however, if properly applied.

Heat a piece of platinum wire in the blue gas flame until it ceases to impart any color to the flame. Dip the wire in a solution of sodium chloride and heat again, observing the color directly and through blue glass. An intense yellow color is imparted to the flame, but this color is cut off by blue glass.

As sodium is widely distributed in small amounts, it is necessary to

be especially careful in cleaning the platinum wire before applying the flame test for it. Also it is necessary to test the original solution for sodium, in separations, because some of the reagents used contain its salts. The flame test for sodium is so delicate that the metal is often found and reported, when its compounds are present only as impurities in a substance.

LITHIUM, $\text{Li}^i = 6.94$

Lithium is a rare metal which is only of importance in a few of its salts, used in medicine. Its compounds are generally colorless and soluble in water.

IMPORTANT COMPOUNDS OF LITHIUM.

Lithium bromide, U. S. P., LiBr .

Lithium carbonate, U. S. P., Li_2CO_3 .

Lithium chloride, LiCl .

TESTS FOR LITHIUM.

Use a separate portion of a solution of a lithium salt, as LiCl , for each of the following tests:

1. Add Na_2CO_3 T.S. to the lithium solution. A white precipitate of lithium carbonate, Li_2CO_3 , is formed if the solution is sufficiently concentrated.

2. Add Na_2HPO_4 T.S. and NaOH T.S. to the lithium solution and boil the mixture. A white precipitate of lithium phosphate, Li_3PO_4 , will form, if the solution is not too dilute.

3. Heat some of the lithium solution on platinum wire in a blue gas flame and observe the color of the flame, directly and through blue glass. The flame is colored a deep red, which is visible through a single piece of blue glass of ordinary thickness, but is not visible through several thicknesses of blue glass.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{LiCl} + \text{Na}_2\text{CO}_3 =$
 $\text{LiBr} + \text{K}_2\text{CO}_3 =$
2. $\text{LiBr} + \text{Na}_2\text{HPO}_4 =$
 $\text{LiCl} + (\text{NH}_4)_2\text{HPO}_4 =$

AMMONIUM, $(\text{NH}_4)^i = 18.042$

Ammonium is a positive compound radical which forms a series of salts that are very similar to the salts of potassium and sodium. All ammonium salts are volatilized or decomposed by heat and most of them are soluble in water.

IMPORTANT AMMONIUM COMPOUNDS.

Ammonia, NH_3 .

Ammonium acetate, $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$.

Ammonium bromide, U. S. P., NH_4Br .

Ammonium carbonate, U.S.P., sal volatile, $\text{NH}_4\text{HCO}_3 + \text{NH}_4\text{NH}_2\text{CO}_2$.

Ammonium chloride, U. S. P., sal ammoniac, NH_4Cl .

Ammonium hydroxide, NH_4OH (in solution only).

Ammonium hypophosphite, N. F., $\text{NH}_4\text{PH}_2\text{O}_2$.

Ammonium iodide, U. S. P., NH_4I .

Ammonium nitrate, NH_4NO_3 .

Ammonium oxalate, $(\text{NH}_4)_2\text{C}_2\text{O}_4\text{H}_2\text{O}$.

Ammonium phosphate, N. F., $(\text{NH}_4)_2\text{HPO}_4 + \text{NH}_4\text{H}_2\text{PO}_4$.

Ammonium sulphate, $(\text{NH}_4)_2\text{SO}_4$.

Ammonium sulphide, $(\text{NH}_4)_2\text{S}$.

Ammonium polysulphide, yellow ammonium sulphide, $(\text{NH}_4)_2\text{S}_x$.

TESTS FOR AMMONIUM.

Use a separate portion of a solution of an ammonium salt, as NH_4Cl , for each of the following tests:

1. Add NaOH T.S. to the ammonium solution until the reaction is alkaline to litmus, and boil the mixture. Ammonia gas, NH_3 , is evolved. Observe the odor of ammonia in the vapor. Moisten a piece of red litmus paper and hold it in the vapor: the color will be changed to blue. Dip a glass rod in concentrated HCl and hold it in the vapor: white fumes of ammonium chloride, NH_4Cl , are formed.

2. Add $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ T.S. to the ammonium solution. A white precipitate of ammonium bi-tartrate, $\text{NH}_4\text{HC}_4\text{H}_4\text{O}_6$, will be formed, if the solution is not too dilute. The precipitate is soluble in alkalis.

3. Acidulate a portion of the ammonium solution with $\text{HC}_2\text{H}_3\text{O}_2$ T.S., add $\text{Na}_3\text{Co}(\text{NO}_2)_6$ T.S. and let stand. A yellow precipitate of ammonium-cobaltic nitrite, $(\text{NH}_4)_3\text{Co}(\text{NO}_2)_6$, will form slowly.

4. Add alkaline HgK_2I_4 T.S. (Nessler's reagent) to the ammonium solution. A brown precipitate of nitrogen dimercuric iodide, NHg_2I , is formed if the solution is concentrated. With dilute solutions of ammonium salts this reagent gives a deep brown color, but no precipitate. Several of the metals interfere with this reaction.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{NH}_4\text{Cl} + \text{NaOH} =$
 $(\text{NH}_4)_2\text{SO}_4 + \text{KOH} =$
 $\text{NH}_3 + \text{HCl} =$
2. $\text{NH}_4\text{Br} + \text{H}_2\text{C}_4\text{H}_4\text{O}_6 =$
 $\text{NH}_4\text{NO}_3 + \text{NaHC}_4\text{H}_4\text{O}_6 =$

3. $\text{NH}_4\text{Cl} + \text{Na}_3\text{Co}(\text{NO}_2)_6 =$
 $\text{NH}_4\text{NO}_3 + \text{Na}_3\text{Co}(\text{NO}_2)_6 =$
 4. $\text{NH}_4\text{Cl} + 2\text{HgK}_2\text{I}_4 + 4\text{KOH} = \text{NHg}_2\text{I} + 7\text{KI} + \text{KCl} + 4\text{H}_2\text{O}$

SAMPLE NUMBER 12.

Analysis of a solution for group 7 of the metals.

The sample may contain compounds of any of the following metals: magnesium, lithium, sodium, potassium, ammonium.

A. Acidulate about 5 mls of the sample with HCl T.S., evaporate the liquid to a small volume on a water-bath, transfer to a porcelain crucible and ignite gently, to volatilize any ammonium compounds. Dissolve the residue in about 10 mls of water in a beaker, adding a few drops of HCl T.S., to obtain a clear solution, if necessary.

B. Add an excess of NaOH T.S. to the solution obtained in *A* and boil. If no precipitate is produced, magnesium is absent. A white precipitate, $\text{Mg}(\text{OH})_2$, shows the presence of magnesium. Filter and test the filtrate.

C. To the clear filtrate obtained in *B*, add Na_2HPO_4 T.S. and boil. If no precipitate is produced, lithium is absent. A white precipitate, Li_3PO_4 , which may appear as no more than a turbidity in the liquid, shows the presence of lithium.

D. Hold the end of a piece of platinum wire in the blue flame of a gas burner until the flame no longer shows a color from the wire. Dip the wire in the original solution and hold it in the flame again.

If the flame is bright yellow when looked at directly but shows no color through blue glass, sodium is present and potassium is absent.

If the flame is violet when looked at directly and through double thick blue glass, potassium is present and sodium is absent.

If the flame is bright yellow when looked at directly and shows a violet color through double thick blue glass, both sodium and potassium are present.

If lithium is present these flame reactions are affected by its deep red flame, but this can be allowed for after experience with the tests described above.

E. To a portion of the original solution add an excess of NaOH T.S. and boil. If no odor of ammonia can be detected and the steam will not turn red litmus paper blue, ammonium is absent. If the steam smells of ammonia, or turns red litmus paper blue, ammonium is present.

SAMPLE NUMBER 13.

Analysis of a solution for all groups of the metals.**Separation of the metals into groups.**

The solution should be neutral or acid in reaction to litmus. If alkaline add HNO_3 to the portion to be examined for metals until the reaction is acid. Add HCl as long as a precipitate is produced and filter.

Precipitate	Filtrate	
Pb, Hg', Ag.	Warm and pass H_2S as long as a precipitate is produced. Filter.	
Examine for metals of group 1 by I.	Precipitate	Filtrate
	Digest with $(\text{NH}_4)_2\text{S}_x$ and filter.	Boil to expel H_2S . Add a few drops of HNO_3 and boil again, then add NH_4Cl and an excess of NH_4OH and boil again. Filter.
	Residue Hg', Pb, Bi, Cu, Cd.	Precipitate Sb, Sn, As, Au, Pt.
	Examine for metals of group 2 by II.	Examine for metals of group 3 by III.
		Precipitate Al, Cr, Fe.
		Examine for metals of group 4 by IV.
		Filtrate
		Pass H_2S as long as a ppt. is produced. Filter.
		Precipitate Zn, Mn, Co, Ni.
		Examine for metals of group 5 by V.
		Filtrate
		Boil to expel H_2S , add an excess of $(\text{NH}_4)_2\text{CO}_3$ and filter.
		Precipitate Ba, Sr, Ca.
		Examine for metals of group 6 by VI.
		Filtrate Mg, Li, Na, K, (NH_4)'
		Examine for metals of group 7 by VII.

I.

Examination of any precipitate produced by HCl in a neutral or acid solution.

Wash the precipitate on the filter with cold water, discarding the washings. Pass a portion of hot water through the washed precipitate on the filter.

Filtrate— PbCl_2	Residue— $\text{HgCl}_2, \text{AgCl}$	
Add H_2SO_4 and cool. White ppt. PbSO_4 , shows	Pass the same portion of NH_4OH through the residue on the filter several times	
	Residue—$\text{NH}_4\text{HgCl} + \text{Hg}$, black, shows	Filtrate, $(\text{NH}_4)_2(\text{AgCl})_2$. Add an excess of HNO_3, white ppt., AgCl, shows
lead	mercurous mercury	silver

II.

Examination of any residue insoluble in $(\text{NH}_4)_2\text{S}_x$ from a precipitate produced by H_2S in an acid liquid, after group 1 of the metals has been removed from a solution.

Wash the residue on the filter, first with an additional portion of $(\text{NH}_4)_2\text{S}_x$ and then with water, and drain, discarding the washings.

Pour a portion of hot HNO_3 through the filter several times.

Residue, HgS	Filtrate—$\text{Pb}(\text{NO}_3)_2$, $\text{Bi}(\text{NO}_3)_3$, $\text{Cu}(\text{NO}_3)_2$, $\text{Cd}(\text{NO}_3)_2$			
Black. Dissolve in nitrohydrochloric acid, boil to expel Cl , dilute with water, filter and add SnCl_2 . A white or gray ppt. $\text{HgCl}_2 + \text{Hg}$ shows	Add conc. H_2SO_4 and boil until white fumes are given off, dilute with H_2O , cool and filter.			
	Precipitate, PbSO_4	Filtrate—$\text{Bi}_2(\text{SO}_4)_3$, CuSO_4, CdSO_4		
	White. Dissolve with $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ and add $\text{K}_2\text{Cr}_2\text{O}_7$. Yellow ppt., PbCrO_4 , shows	Add an excess of NH_4OH and filter.		
		Precipitate, $\text{Bi}(\text{OH})_3$	Filtrate—$\text{Cu}(\text{NH}_4)_2\text{SO}_4$, $\text{Cd}(\text{NH}_4)_2\text{SO}_4$	
		White. Add NaOH and SnCl_2 on filter. Brown or black coloration, Bi_2O_3 , shows	Divide into two portions.	
			To one portion add an excess of $\text{HC}_2\text{H}_3\text{O}_2$ and then $\text{K}_4\text{Fe}(\text{CN})_6$. A red ppt. or coloration, $\text{Cu}_2\text{Fe}(\text{CN})_6$, shows	Pass H_2S through the other portion, first decolorizing with KCN if Cu is present. A yellow ppt., CdS , shows
mercuric mercury	lead	bismuth	copper	cadmium

III.

Examination of the solution in $(\text{NH}_4)_2\text{S}_x$ of any precipitate produced by H_2S in an acid liquid, after group I of the metals has been removed from a solution.

Acidulate the $(\text{NH}_4)_2\text{S}_x$ solution with HCl and collect any precipitate formed on a filter. Transfer the precipitate to a test tube and digest in $(\text{NH}_4)_2\text{CO}_3$ T.S. Filter.

Filtrate—$(\text{NH}_4)_3\text{AsS}_4$	Residue—Sb_2S_3, SnS_2, Au_2S_3, PtS_2			
Add excess of HCl and pass H_2S . A yellow ppt., As_2S_3 , shows	Wash with water. Digest in concentrated HCl . Filter.			
	Filtrate—SbCl_3, SnCl_4	Residue—Au_2S_3, PtS_2		
	Place in H generator. Add H_2O and Zn . Conduct gas into AgNO_3 for three minutes	If light colored Au and Pt are absent. If dark colored, digest in nitrohydrochloric acid. Add H_2O and filter.		
	If black ppt. forms in AgNO_3 , dissolve in HCl and pass H_2S . An orange ppt., Sb_2S_3 , shows	Filter liquid from generator and add few drops to HgCl_2 . A white or gray ppt., $\text{HgCl}_2 + \text{Hg}$, shows	To a part of the filtrate add SnCl_2 and SnCl_4 and let stand. A purple ppt., Au_2O_3 , etc., shows	Neutralize another part of the filtrate with NH_4OH , add alcohol and NH_4Cl . A yellow ppt., $(\text{NH}_4)_2\text{PtCl}_6$, shows
arsenic	antimony	tin	gold	platinum

IV.

 NH_4OH

Examination of any precipitate produced by NH_4OH , after groups 1, 2 and 3 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Boil the precipitate with $NaOH$ for several minutes and filter.

Filtrate, $NaAlO_2$.		Residue— $Cr(OH)_3$, $Fe(OH)_3$.	
Acidulate with HCl , then add an excess of $(NH_4)_2CO_3$ and boil. A white ppt., $Al(OH)_3$, shows		Wash with H_2O and drain, discarding the washings. Transfer a part of the residue to a porcelain crucible cover, add about the same amount of dry KNO_3 and about twice as much dry Na_2CO_3 and ignite until fused. Cool, heat the mass with H_2O and filter.	
		Filtrate, K_2CrO_4 .	
		Acidulate with $HC_2H_3O_2$ and add $Pb(C_2H_3O_2)_2$. A yellow ppt., $PbCrO_4$, shows	
aluminum	chromium	Residue— Fe_2O_3 .	
		Wash on the filter with water, discarding the washings. Dissolve in HCl and add $KCNS$. A red coloration, $Fe(CNS)_3$, shows	
		iron	
		If iron is present test separate portions of the original solution as follows:	
		To one portion add HCl and $K_3Fe(CN)_6$. A dark blue ppt., $Fe_3(Fe(CN)_6)_2$, shows	To another portion add HCl and $KCNS$. A red coloration, $Fe(CNS)_3$, shows
		ferrous iron	ferric iron

V.

Examination of any precipitate produced by H_2S in an alkaline liquid, after groups 1, 2, 3 and 4 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Pass the same portion of HCl through the filter several times.

Filtrate— $ZnCl_2$, $MnCl_2$.		Residue— CoS , NiS .	
Boil to expel H_2S , cool, add excess of $NaOH$ and filter.		If light colored, cobalt and nickel are absent. If dark colored, dissolve in nitrohydrochloric acid, add an excess of $NaOH$, collect the ppt. produced on a filter and wash, discarding the washings. Dissolve the ppt. in $HC_2H_3O_2$, add an excess of KCl and of KNO_3 , let stand for half an hour and filter.	
Filtrate, Na_2ZnO_2 .		Residue, $Mn(OH)_2$.	
Pass H_2S . A white ppt. ZnS , shows		Boil with HNO_3 and let stand. A pink supernatant liquid shows	
zinc	manganese	Precipitate, $K_2Co(NO_2)_6$, yellow, shows	
		Filtrate, $Ni(C_2H_3O_2)_2$. Add an excess of $NaOH$. Light green ppt. $Ni(OH)_2$, shows	
		cobalt	nickel

VI.

Examination of any precipitate produced by $(\text{NH}_4)_2\text{CO}_3$, after groups 1, 2, 3, 4 and 5 of the metals have been removed from a solution.

Wash the precipitate on the filter with water and drain, discarding the washings. Dissolve the washed precipitate in $\text{HC}_2\text{H}_3\text{O}_2$, add an excess of $\text{K}_2\text{Cr}_2\text{O}_7$ and filter.

Precipitate, BaCrO_4	Filtrate— $\text{Sr}(\text{C}_2\text{H}_3\text{O}_2)_2$, $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$.	
	Divide into two portions.	
Yellow shows	To one portion add CaSO_4 , boil and let stand. White ppt. SrSO_4 , shows	To the other portion, add K_2SO_4 , boil, cool and filter, if necessary. To the filtrate add $(\text{NH}_4)_2\text{C}_2\text{O}_4$. White ppt. CaC_2O_4 , shows
barium	strontium	calcium

VII.

Examination of a solution for metals not precipitated with groups 1, 2, 3, 4, 5 or 6.

Add an excess of HCl to the filtrate from group 6 and evaporate the liquid to dryness and ignite. Dissolve the residue in water with HCl . Add an excess of NaOH and boil. A white ppt., $\text{Mg}(\text{OH})_2$, shows

magnesium.

Filter out any $\text{Mg}(\text{OH})_2$, add NaH_2PO_4 and boil. A white ppt., Li_3PO_4 , shows

lithium.

Make a flame test of the original solution on platinum wire.

A yellow flame, not visible through blue glass shows

sodium

A violet flame, visible through double thick blue glass shows

potassium

Add an excess of NaOH to a portion of the original solution and boil. An alkaline vapor, NH_3 , having the odor of ammonia, shows

ammonium

THE ACIDS.

The acid radicals cannot be separated from each other by grouping as is done with the metals, and they must often be tested for separately in the original sample. The reactions of the soluble salts of the most important acids are given here. Some others will be noticed later when the preliminary treatment of solids is given.

Three groups of the acids are recognized, as follows:

Group A.—Acids whose radicals are precipitated by silver nitrate, in the presence of nitric acid; hydrochloric acid, HCl ; hydrobromic acid, HBr ; hydriodic acid, HI ; hydrocyanic acid, HCN ; hydrosulphuric acid, H_2S ; and others.

Group B.—Acids whose radicals are precipitated by barium chloride, from a neutral solution: sulphuric acid, H_2SO_4 ; boric acid, H_3BO_3 ; sulphurous acid, H_2SO_3 ; carbonic acid, H_2CO_3 ; oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$; phosphoric acid, H_3PO_4 , and others.

Group C.—Acids whose radicals are not precipitated in groups A and B: acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$; nitric acid, HNO_3 ; and others.

GROUP A.

Acids whose radicals are precipitated as silver salts by silver nitrate in the presence of nitric acid.

The principal members of this group are hydrochloric acid, HCl ; hydrobromic acid, HBr ; hydriodic acid, HI ; hydrocyanic acid, HCN ; and hydrosulphuric acid, H_2S .

HYDROCHLORIC ACID, HCl .

Muriatic acid.

Spirit of salt.

Hydrochloric acid, U. S. P., 31–33%.

Diluted Hydrochloric acid, U. S. P., 9.5–10.5%.

Hydrochloric acid is a colorless gas with a sharp suffocating odor, which is exceedingly soluble in water, and the name is generally applied to the solution. The acid and its solution are irritant or corrosive poisons when inhaled or swallowed. It is monobasic and its salts are called chlorides, formerly muriates. The whole acid combines with vegetable alkaloids and such salts are called hydrochlorides or hydrochlorates.

The chlorides are generally colorless and most of them are soluble in water. Silver chloride and mercurous chloride are insoluble, and lead chloride is only very slightly soluble; also the basic chlorides of bismuth, antimony and tin are insoluble in water.

IMPORTANT SALTS OF HYDROCHLORIC ACID.

Ammonium chloride, U. S. P., sal ammoniac, NH_4Cl .

Barium chloride, $\text{BaCl}_2\text{H}_2\text{O}$.

Calcium chloride, U. S. P., $\text{CaCl}_2\text{2H}_2\text{O}$.

Ferric chloride, U. S. P., $\text{FeCl}_3\text{6H}_2\text{O}$.

Gold chloride, AuCl_3 .

Magnesium chloride, $\text{MgCl}_2\text{6H}_2\text{O}$.

Mercuric chloride, corrosive mercuric chloride, U. S. P., corrosive sublimate, HgCl_2 .

Mercurous chloride, mild mercurous chloride, U. S. P., calomel, HgCl .

Platinum chloride, PtCl_4 .

Potassium chloride, KCl .

Sodium chloride, U. S. P., salt, NaCl .

Stannic chloride, SnCl_4 .

Stannous chloride, $\text{SnCl}_2\text{2H}_2\text{O}$.

Zinc chloride, U. S. P., ZnCl_2 .

TESTS FOR SALTS OF HYDROCHLORIC ACID.

Use separate portions of a solution of a soluble chloride, as NaCl , for each of the following tests:

1. Add AgNO_3 T.S. to the chloride solution. A curdy white precipitate of silver chloride, AgCl , is formed. The precipitate soon darkens upon exposure to light and is insoluble in acids, but is readily soluble in ammonia water.

2. Add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. to the chloride solution. A white precipitate of lead chloride, PbCl_2 , is formed if the solution is not too dilute. The precipitate is slightly soluble in cold water, and more soluble in hot water.

3. Evaporate a portion of the chloride solution to dryness, or nearly so, add concentrated H_2SO_4 and heat. HCl gas will be given off, which can be recognized by its odor and by the white fumes of NH_4Cl it forms if a glass rod or stopper wet with NH_4OH T.S. is held in it.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{NaCl} + \text{AgNO}_3 =$
 $\text{HCl} + \text{AgNO}_3 =$
 $\text{CaCl}_2 + \text{AgNO}_3 =$

2. $\text{KCl} + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$
 $\text{HCl} + \text{Pb}(\text{NO}_3)_2 =$
 $\text{BaCl}_2 + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$
3. $\text{NaCl} + \text{H}_2\text{SO}_4 =$
 $\text{MgCl}_2 + \text{H}_2\text{SO}_4 =$
 $\text{HCl} + \text{NH}_3 =$

HYDROBROMIC ACID, HBr.

Diluted hydrobromic acid, U. S. P., 9.5–10.5%.

Hydrobromic acid is a gas which resembles hydrochloric acid, but is less stable than that acid. Its salts are called bromides.

The bromides are generally colorless and soluble in water. Silver bromide and mercurous bromide are insoluble and lead bromide is only slightly soluble. All bromides are decomposed by chlorine and bromine is set free.

IMPORTANT SALTS OF HYDROBROMIC ACID.

Ammonium bromide, U. S. P., NH_4Br .
Calcium bromide, U. S. P., $\text{CaBr}_2 \cdot 2\text{H}_2\text{O}$.
Lithium bromide, U. S. P., LiBr .
Potassium bromide, U. S. P., KBr .
Silver bromide, AgBr .
Sodium bromide, U. S. P., NaBr .
Strontium bromide, U. S. P., SrBr_2 .

TESTS FOR SALTS OF HYDROBROMIC ACID.

Use a separate portion of a solution of a bromide, as KBr , for each of the following tests:

1. Add AgNO_3 T.S. to the bromide solution. A yellow-white precipitate of silver bromide, AgBr , will be formed, which slowly darkens upon exposure to light. The precipitate is insoluble in nitric acid, but is slowly soluble in ammonia water.

2. Add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. to the bromide solution. A white precipitate of lead bromide, PbBr_2 , will be formed, which is slightly soluble in water.

3. To a few drops of the bromide solution add a few drops of carbon disulphide, CS_2 , and slowly add chlorine T.S. Bromine will be set free and will dissolve in the CS_2 to make a brown solution. The color will not disappear when the chlorine is in excess.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{KBr} + \text{AgNO}_3 =$
 $\text{CaBr}_2 + \text{AgNO}_3 =$
2. $\text{NaBr} + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$
 $\text{NH}_4\text{Br} + \text{Pb}(\text{NO}_3)_2 =$
3. $\text{KBr} + \text{Cl}_2 =$
 $\text{CaBr}_2 + \text{Cl}_2 =$

HYDRIODIC ACID, HI.

Diluted hydriodic acid, U. S. P., 9.5–10.5 %.

Hydriodic acid is a heavy colorless gas, which is readily soluble in water. It is unstable and readily reacts with oxygen, forming water and setting iodine free when oxidized. The salts of hydriodic acid are called iodides.

Silver iodide, mercurous iodide, mercuric iodide and lead iodide are insoluble in water. The iodides of other important metals are soluble. All iodides are decomposed by chlorine or bromine and iodine is set free.

IMPORTANT SALTS OF HYDRIODIC ACID.

Ammonium iodide, U. S. P., NH_4I .

Arsenous iodide, U. S. P., AsI_3 .

Ferrous iodide, FeI_2 .

Lead iodide, N.F., PbI_2 .

Mercuric iodide, red mercuric iodide, U. S. P., biniodide of mercury, HgI_2 .

Mercurous iodide, yellow mercurous iodide, U. S. P., protoiodide of mercury, HgI .

Potassium iodide, U. S. P., KI .

Silver iodide, AgI .

Sodium iodide, U. S. P., NaI .

Strontium iodide, U. S. P., SrI .

Zinc iodide, ZnI_2 .

TESTS FOR SALTS OF HYDRIODIC ACID.

Use a separate portion of a solution of an iodide, as KI , for each of the following tests:

1. Add AgNO_3 T.S. to the iodide solution. A yellow precipitate of silver iodide, AgI , will be obtained. The precipitate slowly blackens in the light and is insoluble in HNO_3 or NH_4OH .
2. Add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. to the iodide solution. A yellow precipitate of lead iodide, PbI_2 , will be obtained.
3. To a few drops of the iodide solution add about the same

amount of CS_2 , and slowly add chlorine T.S. Iodine will be set free and will dissolve in the CS_2 to make a pink solution. When the chlorine is in excess the iodine will be oxidized to iodic acid, HIO_3 , and the solution will be decolorized.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{KI} + \text{AgNO}_3 =$
 $\text{NH}_4\text{I} + \text{AgC}_2\text{H}_3\text{O}_2 =$
2. $\text{NaI} + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$
 $\text{ZnI}_2 + \text{Pb}(\text{NO}_3)_2 =$
3. $\text{KI} + \text{Cl}_2 =$
 $\text{CaI}_2 + \text{Cl}_2 =$
 $\text{I}_2 + \text{Cl}_2 + \text{H}_2\text{O} = \text{HIO}_3 +$

HYDROCYANIC ACID, HCN.

Prussic acid.

Diluted hydrocyanic acid, U. S. P., 1.9-2.1%.

Hydrocyanic acid is a colorless liquid with an odor resembling that of bitter almonds. It is soluble in water, but the solution slowly decomposes through the influence of light and air. The salts of hydrocyanic acid are called cyanides, formerly prussiates.

The cyanides of the alkali metals, the alkaline earth metals, and mercuric cyanide are soluble in water. The cyanides of other important metals are insoluble. The acid itself and all of its salts are deadly poisons, especially dangerous because their action is so rapid that there is little time for the administration of antidotes. Care should be taken not to inhale the gas evolved when an acid is added to a cyanide.

IMPORTANT SALTS OF HYDROCYANIC ACID.

Mercuric cyanide, $\text{Hg}(\text{CN})_2$.

Potassium cyanide, KCN.

Silver cyanide, AgCN.

Sodium cyanide, U. S. P., NaCN.

TESTS FOR SALTS OF HYDROCYANIC ACID.

Use a separate portion of a solution of a cyanide, as KCN, for each of the following tests:

1. Slowly add AgNO_3 T.S. to the cyanide solution. A white precipitate of silver cyanide, AgCN, will be formed when the AgNO_3 is in excess. Add an excess of NH_4OH T.S. The precipitate will

form a double compound and dissolve. Silver cyanide is insoluble in diluted HNO_3 .

2. To about 2 mls of the cyanide solution in a test-tube, add about the same amount of each of FeSO_4 T.S. and KOH T.S., and warm, when potassium ferrocyanide $\text{K}_4\text{Fe}(\text{CN})_6$ will be formed. Then add two drops of FeCl_3 T.S. and acidulate with HCl T.S. A blue precipitate of ferric ferrocyanide, $\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$, will be formed.

3. To about 2 mls of the cyanide solution in a porcelain evaporating dish, add 2 drops of yellow ammonium sulphide and heat on a water-bath until the liquid is colorless, potassium sulphocyanate, KCNS , being formed. Then acidulate with HCl T.S. and add a drop of FeCl_3 T.S. A red coloration, due to ferric sulphocyanate, $\text{Fe}(\text{CNS})_3$, will be produced.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{KCN} + \text{AgNO}_3 =$
 $\text{HCN} + \text{AgNO}_3 =$
2. $6\text{KCN} + \text{FeSO}_4 = \text{K}_4\text{Fe}(\text{CN})_6 + \text{K}_2\text{SO}_4$
 $\text{FeCl}_3 + \text{K}_4\text{Fe}(\text{CN})_6 =$
3. $\text{KCN} + (\text{NH}_4)_2\text{S}_2 = \text{KCNS} + 2\text{NH}_3 + \text{H}_2\text{S}$
 $\text{FeCl}_3 + \text{KCNS} =$

HYDROSULPHURIC ACID, H_2S

Hydrogen sulphide.

Sulphuretted hydrogen.

Hydrosulphuric acid is a colorless gas having a characteristic disagreeable odor; it is moderately soluble in water. The gas in solution readily oxidizes and deposits sulphur. The salts of hydrosulphuric acid are called sulphides, formerly sulphurets. Many metals readily combine with additional atoms of sulphur to form polysulphides, which yield hydrogen sulphide and sulphur on decomposition.

The sulphides of the alkali metals—potassium, sodium, lithium and ammonium; and of the alkaline earth metals—magnesium, calcium, strontium and barium; are soluble in water. The sulphides of the other important metals are insoluble.

IMPORTANT SALTS OF HYDROSULPHURIC ACID.

Ammonium sulphide, colorless ammonium sulphide, $(\text{NH}_4)_2\text{S}$.

Ammonium polysulphide, yellow ammonium sulphide, $(\text{NH}_4)_2\text{S}_x$.

Antimonous sulphide, Sb_2S_3 .

Barium sulphide, BaS .

Calcium sulphide, CaS .

Ferrous sulphide, FeS .

Lead sulphide, galena, PbS .

Mercuric sulphide, cinnabar, vermilion, HgS .

Sodium sulphide, Na_2S .

Zinc sulphide, ZnS .

TESTS FOR SALTS OF HYDROSULPHURIC ACID.

Use a separate portion of a solution of a sulphide, as Na_2S , for each of the following tests:

1. Add AgNO_3 T.S. to the sulphide solution. A black precipitate of silver sulphide, Ag_2S , will be obtained, which is insoluble in diluted acids.

2. Add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. to the sulphide solution. A black precipitate of lead sulphide, PbS , will be obtained, which is insoluble in diluted acids.

3. To a few drops of the sulphide solution add H_2SO_4 T.S., and warm. Hold a piece of filter paper moistened with $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. in the gas evolved, and observe the odor. Lead sulphide, PbS will be formed and will blacken the paper, and the odor of H_2S will be observed.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Na}_2\text{S} + \text{AgNO}_3 =$
 $\text{CaS} + \text{AgNO}_3 =$
2. $\text{Na}_2\text{S} + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$
 $(\text{NH}_4)_2\text{S} + \text{Pb}(\text{NO}_3)_2 =$
3. $\text{K}_2\text{S} + \text{H}_2\text{SO}_4 =$
 $\text{BaS} + \text{HCl} =$
 $\text{H}_2\text{S} + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$

SAMPLE NUMBER 14.

Analysis of a solution for the important metals and for group A of the acids.

The sample may contain chlorides, bromides, iodides, cyanides or sulphides of any of the important metals.

A. Examine a portion of the sample for metals by the tables on pages 82-85.

B. To about 5 mls of the sample add H_2SO_4 T.S. and heat, observing the odor of the vapor given off. Hold a piece of lead acetate test-paper in the vapor. If the test-paper is not blackened, sulphides are absent. If the odor of H_2S is perceived and the test-paper is blackened, sulphides are present.

C. To about 3 mls of the sample add a small crystal of FeSO_4 and an excess of NaOH T.S. and heat. Then add two drops of FeCl_3 T.S. and acidulate with HCl . If no blue precipitate is obtained, cyanides are absent. A blue precipitate, $\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$, shows the presence of cyanides.

D. To a few drops of the sample, add about the same amount of CS_2 and then slowly add chlorine T.S. until the chlorine is in excess. If the CS_2 is not colored, iodides and bromides are absent. If the CS_2 is colored pink at first and becomes colorless or brown when the chlorine is in excess, iodides are present. If the CS_2 is colored brown when the chlorine is in excess, bromides are present.

E. If sulphides, cyanides, iodides and bromides are absent, acidulate a small portion of the sample with HNO_3 T.S. and add AgNO_3 T.S. If no precipitate is obtained, chlorides are absent. A white precipitate which readily dissolves in NH_4OH T.S. shows the presence of chlorides.

If sulphides or cyanides are present, add an excess of HNO_3 T.S. to a small portion of the sample and boil, under a hood, to expel H_2S or HCN . Then add AgNO_3 T.S. A white precipitate shows the presence of chlorides.

If bromides or iodides are present, add HNO_3 T.S. to about 5 mls of the sample and boil if necessary to expel H_2S or HCN . Add an excess of AgNO_3 T.S. and filter. Pass about 2 mls of NH_4OH T.S. once through the precipitate on the filter. Any silver chloride quickly dissolves as ammonio-silver chloride. Acidulate the filtrate with HNO_3 . A white precipitate shows the presence of chlorides. This test effectually separates chlorides from iodides, but the separation from bromides is not complete, as silver bromide slowly dissolves in ammonia water. To completely separate chlorides from bromides is difficult of accomplishment, and the methods used are beyond the scope of this manual.

GROUP B.

Acids whose radicals are precipitated from a neutral solution as barium salts by barium chloride or barium nitrate.

The principal members of this group are sulphuric acid, H_2SO_4 ; sulphurous acid, H_2SO_3 ; carbonic acid, H_2CO_3 ; oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$; and phosphoric acid, H_3PO_4 .

SULPHURIC ACID, H_2SO_4 .

Oil of vitrol.

Sulphuric acid, U.S.P., 93–95%. Diluted sulphuric acid, U.S.P. 9.5–10.5%.

Sulphuric acid is a heavy, colorless and odorless liquid which is hygroscopic and non-volatile at ordinary temperatures, but may be

distilled unchanged. The concentrated acid attacks and destroys organic matter and inflicts serious burns if it comes in contact with the skin. It has a strong affinity for water and much heat is generated when the strong acid and water are mixed. On this account, the acid should always be poured into the water, when they are being mixed, so that the rise in temperature will be gradual.

Sulphuric acid, being dibasic, forms normal salts called sulphates, and acid salts called bisulphates. The sulphates and bisulphates are generally colorless and soluble in water. Lead sulphate, barium sulphate and strontium sulphate are insoluble, and mercurous sulphate and calcium sulphate are but slightly soluble.

IMPORTANT SALTS OF SULPHURIC ACID.

Aluminum and ammonium sulphate, ammonia alum, $\text{Al}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminum and potassium sulphate, potash alum, $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Aluminum sulphate, $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$.

Ammonium sulphate, $(\text{NH}_4)_2\text{SO}_4$.

Barium sulphate, heavy spar, permanent white, BaSO_4 .

Cadmium sulphate, CdSO_4 .

Calcium sulphate, gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.

Calcium sulphate, anhydrous, plaster of Paris, CaSO_4 .

Chromium potassium sulphate, chrome alum, $\text{CrK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Cobalt sulphate, $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$.

Copper sulphate, blue vitriol, blue stone, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

Ferric ammonium sulphate, ferric alum, $\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Ferric subsulphate, Monsel's salt, $\text{Fe}_4\text{O}(\text{SO}_4)_5$.

Ferric sulphate, iron tersulphate, $\text{Fe}_2(\text{SO}_4)_3$.

Ferrous sulphate, green vitriol, copperas, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

Magnesium sulphate, Epsom salt, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

Manganous sulphate, $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$.

Mercuric sulphate, HgSO_4 .

Mercurous sulphate, Hg_2SO_4 .

Nickel sulphate, NiSO_4 .

Potassium sulphate, K_2SO_4 .

Silver sulphate, Ag_2SO_4 .

Sodium sulphate, Glauber's salt, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$.

Strontium sulphate, SrSO_4 .

Zinc sulphate, white vitriol, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$.

TESTS FOR SALTS OF SULPHURIC ACID.

Use a separate portion of a solution of a sulphate, as Na_2SO_4 , for each of the following tests:

1. Add BaCl_2 T.S. to the sulphate solution. A finely divided

white precipitate of barium sulphate, BaSO_4 , will be obtained, which is insoluble in HCl or HNO_3 .

2. Add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. to the sulphate solution. A white precipitate of lead sulphate, PbSO_4 , will be obtained, which is insoluble in acids.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Na}_2\text{SO}_4 + \text{BaCl}_2 =$
 $\text{MgSO}_4 + \text{Ba}(\text{NO}_3)_2 =$
2. $\text{K}_2\text{SO}_4 + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 =$
 $\text{ZnSO}_4 + \text{Pb}(\text{NO}_3)_2 =$

SULPHUROUS ACID, H_2SO_3 .

Sulphurous acid is formed when its anhydride, SO_2 , is dissolved in water, but the acid is so unstable that it cannot be isolated.

The acid, being dibasic, forms normal salts, called sulphites, and acid salts, called bisulphites. The sulphites and bisulphites of the alkali metals and the bisulphites of the alkaline earth metals are soluble in water. The other metals do not generally form bisulphites and their sulphites are insoluble. Sulphites and bisulphites are decomposed by most acids, the acid anhydride, sulphur dioxide, SO_2 , being generated.

IMPORTANT SALTS OF SULPHUROUS ACID.

Calcium sulphite, $\text{CaSO}_3 \cdot 2\text{H}_2\text{O}$.

Calcium bisulphite, bisulphite of lime, $\text{Ca}(\text{HSO}_3)_2$.

Sodium sulphite, $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$.

Exsiccated sodium sulphite, U. S. P., Na_2SO_3 .

Sodium bisulphite, NaHSO_3 .

TESTS FOR SALTS OF SULPHUROUS ACID.

Use a separate portion of a solution of a sulphite, as Na_2SO_3 , for each of the following tests:

1. Add BaCl_2 T.S. to the sulphite solution, followed by an excess of HCl and then by a few drops of nitrohydrochloric acid. A white precipitate of barium sulphite, BaSO_3 , will be obtained, which will dissolve in the HCl ; and a precipitate of barium sulphate, BaSO_4 , will be formed upon adding the nitrohydrochloric acid.

2. Add H_2SO_4 T.S. to a small quantity of the sulphite solution and heat the liquid, observing the odor of the gas evolved. The characteristic choking odor is that of sulphur dioxide, SO_2 . The bleaching effect of the gas on organic coloring matter may also be obtained, most easily by holding a colored flower, as a violet, in the gas.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Na}_2\text{SO}_3 + \text{BaCl}_2 =$
 $\text{Ca}(\text{HSO}_4)_2 + \text{Ba}(\text{NO}_3)_2 =$
2. $\text{Na}_2\text{SO}_3 + \text{H}_2\text{SO}_4 =$
 $\text{NaHSO}_3 + \text{HCl} =$

CARBONIC ACID H_2CO_3 .

Carbonic acid appears to be formed when carbon dioxide is dissolved in water, as in "soda water." The compound, however, is so unstable that it cannot be isolated. The acid forms normal salts called carbonates, and acid salts called bicarbonates. Carbonates and bicarbonates are decomposed by nearly all other acids, yielding water and carbon dioxide. The carbonates of the alkali metals are soluble in water. All other carbonates are insoluble in water, though several of them will form unstable bicarbonates and dissolve in water containing carbon dioxide in solution.

IMPORTANT SALTS OF CARBONIC ACID.

Ammonium carbonate, U. S. P., ammonium bicarbonate + ammonium carbamate, $\text{NH}_4\text{HCO}_3 + \text{NH}_4\text{NH}_2\text{CO}_2$.

Calcium carbonate, limestone, marble, chalk, prepared chalk, U. S. P., precipitated calcium carbonate, U. S. P., CaCO_3 .

Ferrous carbonate, FeCO_3 .

Lead sub-carbonate, white lead, $(\text{PbCO}_3)_2\text{Pb}(\text{OH})_2$.

Lithium carbonate, U. S. P., Li_2CO_3 .

Magnesium carbonate, magnesite, MgCO_3 .

Magnesium carbonate, U. S. P., magnesia alba, $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$.

Potassium carbonate, U. S. P., potash, salt of tartar, K_2CO_3 .

Potassium bicarbonate, U. S. P., saleratus, KHCO_3 .

Sodium carbonate, soda, washing soda, $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.

Monohydrated sodium carbonate, U. S. P., $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$.

Sodium bicarbonate, U. S. P., baking soda, NaHCO_3 .

Zinc carbonate, calamine, ZnCO_3 .

TESTS FOR SALTS OF CARBONIC ACID.

Use a separate portion of a solution of a carbonate, as Na_2CO_3 , for each of the following tests:

1. Add BaCl_2 T.S. to the carbonate solution. A white precipitate of barium carbonate, BaCO_3 , will be formed, which is soluble in acids.

2. Add CaCl_2 T.S. to the carbonate solution. A white precipitate of calcium carbonate, CaCO_3 , will be formed, which is soluble in acids.

3. To a portion of the carbonate solution contained in a test-tube provided with a delivery tube, add H_2SO_4 T.S. and conduct the gas generated into lime water, $\text{Ca}(\text{OH})_2$. The carbonate will be decomposed, yielding carbon dioxide, CO_2 , and this will precipitate calcium carbonate, CaCO_3 , from the lime water.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Na}_2\text{CO}_3 + \text{BaCl}_2 =$
 $\text{KHCO}_3 + \text{Ba}(\text{NO}_3)_2 =$
2. $\text{K}_2\text{CO}_3 + \text{CaCl}_2 =$
 $(\text{NH}_4)_2\text{CO}_3 + \text{CaSO}_4 =$
3. $\text{Na}_2\text{CO}_3 + \text{H}_2\text{SO}_4 =$
 $\text{NaHCO}_3 + \text{HCL} =$
 $\text{Ca}(\text{OH})_2 + \text{CO}_2 =$

SAMPLE NUMBER 15.

Analysis of a solution for the important metals and for the radicals of sulphuric, sulphurous and carbonic acids.

The sample may contain any of the important metals, as sulphates, sulphites or carbonates.

A. Examine a portion of the sample for metals by the tables on pages 82-85.

B. To a portion of the sample, add HCl until the reaction is acid and then add BaCl_2 T.S. If no precipitate is produced sulphates are absent. A white precipitate shows the presence of sulphates.

C. To a portion of the sample, contained in a test-tube provided with a delivery tube, add H_2SO_4 T.S., and heat. If a gas is evolved, observe its odor and pass it through lime water. If there is no effervescence of an odorous gas and the lime water is not rendered turbid, sulphites and carbonates are absent. If there is an effervescence of a gas having the odor of burning sulphur, sulphites are present. If there is an effervescence of an odorless gas which renders lime water turbid, carbonates are present. If the odor of burning sulphur is observed and the lime water is rendered turbid, both sulphites and carbonates are present.

OXALIC ACID, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$.

Oxalic acid is an organic acid, whose salts are frequently used as reagents. It is a soluble crystalline solid. The acid is dibasic and forms normal and acid salts, called oxalates, which are generally colorless. The acid and its salts are poisonous. The oxalates of the alkali metals and of chromium are soluble in water. The

oxalates of all other important metals are insoluble in water, but will dissolve in mineral acids, if the metals present form soluble salts with the acids.

IMPORTANT SALTS OF OXALIC ACID.

Ammonium oxalate, $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$.

Calcium oxalate, $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$.

Cerium oxalate, $\text{Ce}_2(\text{C}_2\text{O}_4)_3 \cdot 10\text{H}_2\text{O}$.

Potassium acid oxalate, salt of lemon, $\text{KHC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$.

Sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$.

TESTS FOR SALTS OF OXALIC ACID.

Use a separate portion of a solution of an oxalate, as $(\text{NH}_4)_2\text{C}_2\text{O}_4$, for each of the following tests:

1. Add BaCl_2 T.S. to the oxalate solution. A white precipitate of barium oxalate, BaC_2O_4 , will be formed; the precipitate is soluble in acids which form soluble salts with barium, including acetic acid.
2. Add CaCl_2 T.S. to the oxalate solution. A white precipitate of calcium oxalate, CaC_2O_4 , will be formed, which is soluble in mineral acids but not in acetic acid.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $(\text{NH}_4)_2\text{C}_2\text{O}_4 + \text{BaCl}_2 =$
 $\text{Na}_2\text{C}_2\text{O}_4 + \text{Ba}(\text{NO}_3)_2 =$
2. $\text{K}_2\text{C}_2\text{O}_4 + \text{CaCl}_2 =$
 $\text{KHC}_2\text{O}_4 + \text{CaSO}_4 =$

PHOSPHORIC ACID, H_3PO_4 .

Phosphoric acid, U.S.P., 85-88%. Dilute phosphoric acid, U.S.P., 9.5-10.5%.

Orthophosphoric acid.

Orthophosphoric acid is a colorless deliquescent crystalline solid commonly seen as a syrupy solution in water and whose salts are the phosphates. It is not volatile but when heated loses water and changes, first to pyrophosphoric acid, $\text{H}_4\text{P}_2\text{O}_7$, and then to metaphosphoric acid, HPO_3 .

Orthophosphoric acid is tri-basic and forms normal salts and two series of acid salts. Salts in which only two of the three hydrogen atoms are replaced by metals, as Na_2HPO_4 , are common, and are frequently called phosphates. The phosphates of the alkali metals and the acid phosphates of a few other metals are soluble in water. All other phosphates are insoluble.

IMPORTANT SALTS OF PHOSPHORIC ACID.

Ammonium phosphate, $(\text{NH}_4)_2\text{HPO}_4$.

Calcium phosphate, $\text{Ca}_3(\text{PO}_4)_2$.

Ferric phosphate, FePO_4 .

Sodium phosphate, U. S. P., $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$.

TESTS FOR SALTS OF PHOSPHORIC ACID.

Use a separate portion of a solution of a phosphate, as Na_2HPO_4 , for each of the following tests:

1. Add BaCl_2 T.S. to the phosphate solution. A white precipitate of barium phosphate, BaHPO_4 , will form, which is soluble in acids.

2. To the phosphate solution add CaCl_2 T.S., followed by an excess of $\text{HC}_2\text{H}_3\text{O}_2$ T.S. A white precipitate of calcium phosphate, CaHPO_4 , will be obtained, which will dissolve in the acetic acid.

3. To the phosphate solution add NH_4Cl T.S., NH_4OH T.S. and MgSO_4 T.S. in the order given. A white precipitate of magnesium ammonium phosphate, MgNH_4PO_4 , will form, which is soluble in acids.

4. To about 2 mls of $(\text{NH}_4)_2\text{MoO}_4$ T.S. add a few drops of the phosphate solution and let stand. A yellow precipitate of ammonium phosphomolybdate, $(\text{NH}_4)_3\text{PO}_4(\text{MoO}_3)_{12}$, will form slowly. The precipitate is soluble in alkalis.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{Na}_2\text{HPO}_4 + \text{BaCl}_2 =$
 $(\text{NH}_4)_2\text{HPO}_4 + \text{Ba}(\text{NO}_3)_2 =$
2. $\text{K}_2\text{HPO}_4 + \text{CaCl}_2 =$
 $\text{CaHPO}_4 + \text{HC}_2\text{H}_3\text{O}_2 = \text{Ca}(\text{H}_2\text{PO}_4)_2 +$
3. $\text{NaHPO}_4 + \text{NH}_4\text{OH} + \text{MgSO}_4 =$
 $(\text{NH}_4)_2\text{HPO}_4 + \text{MgCl}_2 =$
4. $\text{Na}_2\text{HPO}_4 + 12(\text{NH}_4)_2\text{MoO}_4 + 23\text{HNO}_3$
 $= (\text{NH}_4)_3\text{PO}_4(\text{MoO}_3)_{12} + 21\text{NH}_4\text{NO}_3 + 2\text{NaNO}_3 + 12\text{H}_2\text{O}.$

SAMPLE NUMBER 16.**Analysis of a solution for the principal metals and for the radicals of oxalic and phosphoric acids.**

The sample may contain any of the important metals as oxalates or phosphates.

A. Add NaOH T.S. to a portion of the sample until the reaction is alkaline, then $\text{HC}_2\text{H}_3\text{O}_2$ T.S. until the reaction is acid, then CaCl_2 T.S. and let stand. If no precipitate is produced oxalic acid is

absent. A white precipitate, which may be formed slowly, shows the presence of oxalates.

B. To about 3 mls of $(\text{NH}_4)_2\text{MoO}_4$ T.S. in a test-tube add a few drops of the sample and let stand. If no precipitate is formed phosphates are absent. A yellow precipitate, slowly formed, shows the presence of phosphates.

About 10 mls of the sample should be examined for the metals by the tables on pages 82-85, with the following changes if oxalates or phosphates, or both, are present.

C. If oxalates are present evaporate the filtrate from the second group to a small volume, transfer to a porcelain crucible, add concentrated HNO_3 and gently ignite, which treatment will destroy oxalic acid. Then dissolve the residue in HCl T.S. and proceed with the examination for group 3 of the metals.

D. If phosphates are present concentrate the filtrate from the second group of metals to about 5 mls, or use the solution obtained in *C* after destroying oxalic acid, if present. Mix with three times its volume of $(\text{NH}_4)_2\text{MoO}_4$ T.S., warm the mixture and let it stand for some time. Filter and test the filtrate. Evaporate the filtrate nearly to dryness to expel nitric acid. Dilute with about 10 mls of water, add a few drops of HCl and pass H_2S through the solution to precipitate the excess of molybdenum, then filter and proceed with the examination for group 3 of the metals, etc.

GROUP C.

Acids whose radicals are not precipitated by any group reagent.

The principal members of this group are boric acid, H_3BO_3 ; acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$; and nitric acid, HNO_3 .

BORIC ACID, H_3BO_3 .

Boric acid, U. S. P.

Boracic acid.

Orthoboric acid.

Boric acid is a crystalline solid which is moderately soluble in water. The acid decomposes when heated, forming successively metaboric acid, HBO_2 ; pyroboric acid or tetraboric acid, $\text{H}_2\text{B}_4\text{O}_7$; and finally the acid anhydride, boron trioxide, B_2O_3 . The salts of ortho-boric acid are unstable and seldom seen. The borates, generally, are the salts of tetraboric acid or metaboric acid, but when decomposed in the presence of water they yield orthoboric acid. The borates are decomposed by nearly all other acids. The borates of the alkali metals are soluble in water. The borates of other metals are insoluble in water but many of them become soluble in the presence of an excess of boric acid.

IMPORTANT SALTS OF BORIC ACID.

Glyceryl borate, boroglycerin, $C_3H_5BO_3$.

Sodium borate, U. S. P., sodium tetraborate, sodium pyroborate, borax, $Na_2B_4O_7 \cdot 10H_2O$.

TESTS FOR SALTS OF BORIC ACID.

Use a separate portion of a solution of a borate, as $Na_2B_4O_7$, for each of the following tests:

1. Add $BaCl_2$ T.S. to the borate solution. A white precipitate of barium metaborate, $Ba(BO_2)_2$, will form if the solution is not too dilute. The precipitate is soluble in an excess of the reagent and in HNO_3 or HCl .

2. Add H_2SO_4 T.S. to a portion of borate solution in a porcelain evaporating dish; then add about twice the volume of alcohol, stir and ignite. The outer mantle of the flame will show a green color. Before applying this test it is necessary to remove copper and barium from the solution, if they are present, by H_2S and H_2SO_4 , respectively.

3. Add HCl T.S. to the borate solution, dip a piece of turmeric paper in the liquid and dry the paper. The paper will be colored red-brown, changing to red upon drying.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $Na_2B_4O_7 + BaCl_2 + H_2O = Ba(BO_2)_2 + Na_2B_4O_7 + Ba(NO_3)_2 + H_2O =$
2. $Na_2B_4O_7 + H_2SO_4 + H_2O = H_3BO_3 +$
3. $Na_2B_4O_7 + HCl + H_2O = H_3BO_3 +$

ACETIC ACID, $HC_2H_3O_2$.

Acetic acid, U. S. P., 36–37%.

Diluted acetic acid, U.S.P., 5.7–6.3%. Glacial acetic acid, U.S.P., 99+%.

Acetic acid is a colorless liquid with a characteristic pungent odor. The pure acid corrodes the skin and freezes to a glass-like solid at temperatures below 15° . The diluted acid has about the strength of vinegar, from which it was first prepared.

The acid is monobasic and its salts are called acetates. All the normal acetates are soluble in water, but insoluble basic acetates are formed by several of the metals.

IMPORTANT SALTS OF ACETIC ACID.

Ammonium acetate, $NH_4C_2H_3O_2$.

Copper acetate, $Cu(C_2H_3O_2)_2 \cdot H_2O$.

Copper aceto-arsenite, paris green, $Cu(C_2H_3O_2)_2 + 3Cu(AsO_2)_2$.

Copper subacetate, verdigris, $\text{Cu}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$.

Ferric acetate, $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3$.

Lead acetate, U. S. P., sugar of lead, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$.

Lead sub-acetate, $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$.

Potassium acetate, U. S. P., $\text{KC}_2\text{H}_3\text{O}_2$.

Sodium acetate, U. S. P., $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$.

Zinc acetate, U. S. P., $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$.

TESTS FOR SALTS OF ACETIC ACID.

Use a separate portion of a solution of an acetate, as $\text{NaC}_2\text{H}_3\text{O}_2$, for each of the following tests:

1. To a portion of the acetate solution add H_2SO_4 T.S., boil the mixture and observe the odor of the vapor, which will be that of the acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$, set free.

2. To about 2 mls of the acetate solution, add a few drops of alcohol, $\text{C}_2\text{H}_5\text{OH}$, and about 1 mil of H_2SO_4 T.S. and heat, observing the odor of the vapor. The characteristic fruity odor of ethyl acetate, $\text{C}_2\text{H}_5\text{C}_2\text{H}_3\text{O}_2$ will be observed.

3. Add FeCl_3 T.S. to the acetate solution which should be neutral or only slightly acid in reaction, and boil the mixture. Ferric acetate, $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3$, is formed and colors the liquid red. Upon boiling a brown-red precipitate of a basic ferric acetate, $\text{FeOC}_2\text{H}_3\text{O}_2$, will be obtained.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $\text{NaC}_2\text{H}_3\text{O}_2 + \text{H}_2\text{SO}_4 =$
 $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{HCl} =$
2. $\text{NaC}_2\text{H}_3\text{O}_2 + \text{H}_2\text{SO}_4 + \text{C}_2\text{H}_5\text{OH} = \text{NaHSO}_4 +$
3. $\text{KC}_2\text{H}_3\text{O}_2 + \text{FeCl}_3 =$
 $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{FeCl}_3 =$
 $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3 + \text{H}_2\text{O} =$

NITRIC ACID, HNO_3 .

Aqua fortis.

Nitric acid, U.S.P., 67-69%.

Nitric acid is a colorless fuming liquid which is unstable and difficult to prepare in a pure state. The official acid is a concentrated solution and is a dangerously corrosive and poisonous liquid. It is a strong acid and also a powerful oxidizing agent. "Fuming nitric acid" is brown in color and is a solution of nitrogen peroxide, NO_2 , in concentrated nitric acid.

The acid is monobasic and its salts are called nitrates. All normal nitrates are soluble in water.

IMPORTANT SALTS OF NITRIC ACID.

Ammonium nitrate, NH_4NO_3 .
 Barium nitrate, $\text{Ba}(\text{NO}_3)_2$.
 Bismuth nitrate, $\text{Bi}(\text{NO}_3)_3$.
 Bismuth sub-nitrate, U. S. P., approximately $\text{Bi}(\text{OH})_2\text{NO}_3$.
 Ferric nitrate, $\text{Fe}(\text{NO}_3)_3$.
 Lead nitrate, $\text{Pb}(\text{NO}_3)_2$.
 Mercuric nitrate, $\text{Hg}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$.
 Potassium nitrate, U. S. P., saltpetre, nitre, KNO_3 .
 Silver nitrate, U. S. P., lunar caustic, AgNO_3 .
 Sodium nitrate, Chili saltpetre, NaNO_3 .
 Strontium nitrate, $\text{Sr}(\text{NO}_3)_2$.

TESTS FOR SALTS OF NITRIC ACID.

Use a separate portion of a solution of a nitrate, as KNO_3 , for each of the following tests:

1. To a portion of the nitrate solution add an equal volume of H_2SO_4 T.S. and a small amount of metallic copper and heat the mixture. The nitrate is decomposed and gaseous nitric oxide, NO , is formed. This oxidizes in the air and the brown gas, nitrogen peroxide, NO_2 , is obtained.

2 **BROWN RING TEST.** Mix about 1 mil of the nitrate solution with about the same volume of concentrated H_2SO_4 , and cool the mixture. Then carefully pour some freshly made FeSO_4 T.S. down the side of the test-tube so that the liquids do not mix and let stand. The liberated nitric acid will oxidize some of the iron to form ferric sulphate and this will give a brown ring between the two layers.

The test may also be applied by mixing the solution to be tested with FeSO_4 T.S. and superimposing the mixture on concentrated H_2SO_4 in a test-tube.

COMPLETE AND BALANCE THE FOLLOWING EQUATIONS:

1. $2\text{KNO}_3 + 4\text{H}_2\text{SO}_4 + 3\text{Cu} = \text{K}_2\text{SO}_4 + 3\text{CuSO}_4 + 4\text{H}_2\text{O} + 2\text{NO}$
 $2\text{NO} + \text{O}_2 = 2\text{NO}_2$
2. $\text{KNO}_3 + \text{H}_2\text{SO}_4 =$
 $6\text{FeSO}_4 + 3\text{H}_2\text{SO}_4 + 2\text{HNO}_3 = 3\text{Fe}_2(\text{SO}_4)_3 + 4\text{H}_2\text{O} + 2\text{NO}$

SAMPLE NUMBER 17.

Analysis of a solution for the principal metals and for the radicals of boric, acetic and nitric acids.

The sample may contain any of the important metals, as borates, acetates or nitrates.

A. Examine a portion of the sample for metals by the tables on pages 82-85.

B. To a portion of the sample add HCl T.S., dip a piece of turmeric paper in the liquid and dry the paper. If the color of the paper is unchanged borates are absent. If the paper is turned red-brown, changing to dark red upon drying, borates are present.

C. To a portion of the sample add a few drops of alcohol followed by an excess of H_2SO_4 T.S., heat the liquid and observe the odor of its vapor. If no odor of ethyl acetate can be observed, acetates are absent. If the odor of ethyl acetate is observed, acetates are present.

D. Mix some of the sample with about the same volume of concentrated H_2SO_4 in a test-tube, cool the liquid, superimpose FeSO_4 T.S. on it and let stand. If no brown ring forms at the junction of the two liquids, nitrates are absent. If a brown ring forms at the junction of the liquids, nitrates are present.

SAMPLE NUMBER 18.

Analysis of a solution for the important metals and acids.

The sample may contain any of the important metals as salts of any of the acids studied.

A. Add NaOH T.S. to a portion of the sample until the reaction is alkaline, then $\text{HC}_2\text{H}_3\text{O}_2$ T.S. until the reaction is acid, then CaCl_2 T.S. and let stand. If no precipitate is produced, oxalates are absent. A white precipitate, which may be formed slowly, shows the presence of oxalates.

B. To about 3 mls of $(\text{NH}_4)_2\text{MoO}_4$ T.S. in a test-tube add a few drops of the sample, warm and let stand. If no precipitate forms, phosphates are absent. A yellow precipitate, slowly formed, shows the presence of phosphates. *10 minutes*

C. Examine a portion of the sample for metals by the tables on pages 82-85, removing oxalates and phosphates if present as directed on pages 99-100.

D. To about 5 mls of the sample add an excess of H_2SO_4 T.S. and heat. If a gas is given off observe its odor, hold a piece of paper wet with $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ T.S. in the gas and pass some of it through lime water.

A gas having the odor of burning sulphur shows the presence of sulphites.

A gas having the odor of H_2S and which blackens $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ shows the presence of sulphides.

An odorless gas which renders lime water turbid shows the presence of carbonates.

E. Add a slight excess of HNO_3 T.S. to a portion of the sample, followed by AgNO_3 T.S. If no precipitate is produced, the acids of

group A are absent: pass to paragraph I. If a precipitate is produced, proceed by paragraph F, remembering that sulphides, if present, will have been detected in D.

F. To about 3 mls of the sample add a small crystal of FeSO_4 and an excess of NaOH T.S. and heat. Then add two drops of FeCl_3 T.S. and acidulate with HCl . A blue precipitate, $\text{Fe}_4(\text{Fe}(\text{CH})_6)_3$, shows the presence of cyanides.

G. To a few drops of the sample, add about the same amount of CS_2 and then slowly add chlorine T.S. until the chlorine is in excess. If the CS_2 is not colored, iodides and bromides are absent. If the CS_2 is colored pink at first and becomes colorless or brown when the chlorine is in excess, iodides are present. If the CS_2 is colored brown when the chlorine is in excess, bromides are present.

H. If sulphides, cyanides, iodides and bromides are absent, acidulate a small portion of the sample with HNO_3 T.S. and add AgNO_3 T.S. A white precipitate which readily dissolves in NH_4OH T.S. shows the presence of chlorides.

If sulphides or cyanides are present, add an excess of HNO_3 T.S. to a small portion of the sample and boil, under a hood, to expel H_2S or HCN . Then add AgNO_3 T.S. A white precipitate shows the presence of chlorides.

If bromides or iodides are present, add HNO_3 T.S. to about 5 mls of the sample and boil if necessary to expel H_2S or HCN . Add an excess of AgNO_3 T.S. and filter. Pass about 2 mls of NH_4OH T.S. once through the precipitate on the filter. Any silver chloride quickly dissolves as ammonio-silver chloride. Acidulate the filtrate with HNO_3 . A white precipitate shows the presence of chlorides.

I. Acidulate a portion of the sample, add HCl T.S., and add BaCl_2 T.S. A white precipitate shows the presence of sulphates. If a precipitate is formed upon adding HCl T.S., it should be filtered out and the BaCl_2 T.S. should be added to the clear filtrate.

J. Add H_2SO_4 T.S. and an equal volume of alcohol to a portion of the sample contained in a porcelain evaporating dish and set fire to the alcohol. If the flame is colored green, borates are present.

Barium and copper, if present, must be removed before applying this test for boric acid.

K. Add an excess of H_2SO_4 T.S. and a few drops of alcohol to a portion of the sample, boil and observe the odor of the vapor. An odor of ethyl acetate, $\text{C}_2\text{H}_5\text{C}_2\text{H}_3\text{O}_2$, shows the presence of acetates.

L. Mix a portion of the sample with about the same volume of concentrated H_2SO_4 , superimpose FeSO_4 T.S. on the liquid and let stand. A brown zone between the two liquids shows the presence of nitrates.

SOLIDS.

Solid substances must generally be put into solution before they can be examined for bases and acids, and the tests must be applied to the solutions obtained; but the behavior of the solid, alone or with reagents, will often give information leading to the identification of its constituents, and various methods for the examination of solids have been devised. The method given here is aimed at bringing the solid into solution by the use of various solvents. Incidentally, some information of analytical value may be obtained by observing the directions closely.

SAMPLES NUMBERS 19 AND 20.

Qualitative analysis of a solid.

The sample may contain any of the bases or acids considered in the course. Interfering organic substances are absent.

A. The sample should be finely powdered with a mortar and pestle, or otherwise, if it is not already in a powdered condition.

B. Place a small portion of the powdered sample in a small dry test-tube and heat in a gas flame, carefully observing its behavior and the color and odor of any gas or vapor given off, also any condensation of vapors on the cool portion of the tube.

If no apparent change occurs in the solid and no odor is observed, and there is no condensation of vapors, then organic matter, volatile bodies and water of crystallization are absent.

If the solid blackens and there is an odor of something burning, organic matter is present. If organic matter were present it would be necessary to destroy it, by methods which will be described later, as it would interfere with the analytical scheme.

Compounds of some of the metals change color or fuse when heated, but the changes are not significant enough to be of much analytical value. Such metals, if present, will be found when the groups are separated.

If the substance gives off a vapor which condenses on the cool part of the tube, volatile substances are contained in the solid, or are formed upon heating it. These may include water; any compounds of ammonium, arsenic, antimony and mercury; or some compounds of sulphur, iron and iodine. The water may generally be recognized as minute drops and the other substances will be found in their proper places when the scheme of analysis is applied.

If a gas is evolved, observe its odor and test it by holding a splinter of wood or a cord with a spark at the end in it, and by passing some of it through lime water. H_2S , SO_2 , NH_3 , NO_2 and some other gases may be recognized by their odors. If the gas makes a spark glow more vividly it contains more oxygen than the air and this indicates the presence of nitrates, chlorates, bromates, iodates or peroxides. If organic matter is absent and the gas renders lime water turbid, it contains carbon dioxide and the presence of carbonates is indicated.

C. Boil about 1 gram of the finely divided sample with about 15 mls of water in a beaker. If the solid does not completely dissolve allow the residue to settle and decant the water through a filter. Evaporate a few drops of the filtrate to dryness on a watch-glass. If no appreciable deposit is left on the watch-glass the sample is insoluble in water. In such a case proceed with paragraph *D*, discarding the rest of the filtrate. If any appreciable deposit is left on the watch-glass, the rest of the filtrate, or the solution if the sample all dissolved in water, should be examined for the bases by the tables on pages 82-85 and for acids as directed on pages 92, 97, 99 and 103.

D. If there is any residue insoluble in water from *C*, wash it with water by decantation and filtration and allow to drain, discarding the washings. Transfer two small portions of the moist powder to test-tubes and boil one with HCl T.S. and the other with HNO_3 T.S., observing which of the acids appears to be the better solvent for the powder.

If there is an effervescence upon adding the HCl T.S. to the powder, observe the color and odor of the gas and pass some of it through lime water. A greenish-yellow gas indicates a chlorate, a brown gas indicates a nitrate, and a purple gas indicates an iodide. Gases with characteristic odors are yielded by salts of H_2S , H_2SO_3 , HBr , HI , HCN and $\text{HC}_2\text{H}_3\text{O}_2$. If the gas renders lime water turbid, carbonates are indicated. It should be remembered that chlorine might be generated from the HCl used, if certain oxidizing agents are present.

Boil the rest of the moist powder with about 10 mls of the acid which appears to dissolve it the better. If neither acid appears to dissolve any of the powder, or both dissolve it entirely, use HCl T.S. in preference to HNO_3 T.S. If there is any residue insoluble in the acids, allow it to settle and decant the supernatant liquid through the filter used for the water solution and test a few drops for dissolved matter by evaporating on a watch-glass as before. If any appreciable deposit is left on the watch-glass, the rest of the filtrate, or the solution if all the residue dissolved in the acid, should be examined for bases by the tables on pages 82-85, and for acids as directed on pages 92, 97, 99 and 103.

E. If there is any residue insoluble in HCl or HNO_3 , from *D*, it should be washed by decantation and filtration as before and digested with about 5 mls of nitro-hydrochloric acid. Then add about 10 mls of water and filter, testing the filtrate for dissolved matter by evaporation on a watch-glass and examine for bases and acids, if any appreciable amount of the solid goes into solution.

F. If sufficient quantities of the solvents have been used and there is still some residue left from *E*, it consists of insoluble sulphates, chlorides or silicates. To decompose these transfer the washed and drained residue to a porcelain crucible, mix it with about twice its bulk of dry Na_2CO_3 and heat to fusion. Then allow the mass to cool and boil it with about 10 mls of water. Filter the liquid and examine the filtrate for acids by paragraph *G*. Dissolve any residue in HCl or HNO_3 , as in paragraph *D* and examine the solution for bases by the tables on pages 82-85.

G. To the filtrate from *F* add an excess of HNO_3 T.S. and boil. If no precipitate is formed, silicates are absent. A white gelatinous precipitate shows the presence of silicates. If a precipitate is formed filter the liquid. Divide the liquid or filtrate into two portions.

To one portion add BaCl_2 T.S. If no precipitate is formed, sulphates are absent. A white precipitate shows the presence of sulphates.

To the other portion, add AgNO_3 T.S. If no precipitate is formed, chlorides are absent. A white precipitate, readily soluble in ammonia water, shows the presence of chlorides.

QUALITATIVE EXAMINATION OF OFFICIAL INORGANIC CHEMICALS.

In speaking of the *purity* of a chemical substance we refer to the kinds and amounts of other substances that it contains. These other substances are called *impurities*.

To manufacture an absolutely pure chemical compound, it would be necessary to use nothing but absolutely pure substances and not allow the compound to come into contact with anything that would contaminate it at any step in the process of manufacture. Such ideal conditions are seldom or never found, as natural substances are not often chemically pure and most chemicals will mix with more or less of contaminating substances during their processes of manufacture. Compounds approximating chemical purity, however, are obtained by working as nearly as possible under the ideal conditions, or by purifying compounds of a lower degree of purity. In general, water is considered as an impurity only to the extent that it affects the strength or concentration of a chemical.

Many grades of the same chemical are manufactured. The lowest commercial grade is commonly said to be *technically* pure and contains enough of the chemical to give the substance identity, but may contain considerable amounts of impurities. Chemicals of this grade are made in large quantities and are used in many kinds of manufacturing work. The highest grade of a chemical is generally labelled *chemically pure* and is as nearly absolutely pure as can be obtained under manufacturing conditions. Such chemicals are relatively high in price and are only used when such a high grade of purity is essential, as in the preparation of reagents for analytical work.

For medicinal use, chemicals are required to be free from appreciable amounts of objectionable impurities and of proper strength, but the United States *Pharmacopæia* and the *National Formulary* do not require that they be of chemically pure grade in many cases, as the excessive cost of such chemicals is not warranted, provided that the impurities are not harmful or excessive in amount. A similar rule can properly be applied to unofficial chemicals. The allowable amount of impurities is stated in the *purity rubric*, which is a part of the definition of official chemicals and states the minimum strength required for concentrated chemicals and the minimum and maximum strengths allowed for solutions of chemicals.

Qualitative tests on official chemicals are of two kinds, namely, *identity tests* and *purity tests*, these being used, respectively, to establish the identity of the substance under examination, and to ascertain the presence or absence of excessive amounts of objectionable impurities. The strengths of different chemicals are determined by assays, which belong to the domain of quantitative analysis.

The directions for work on samples 21–25 are taken from the United States *Pharmacopæia*, with some omissions and modifications, made to keep the work within the scope of an elementary course in qualitative analysis.

SAMPLE NUMBER 21.

Examination of granulated ferrous sulphate for identity and purity.
(U. S. P., pp. 172 and 170.)

The tests should be carried out as directed, the following report form being copied and filled out as the results of each test are obtained.

Name..... Date.....

Record of work and report on sample No. 21.

Qualitative examination of granulated ferrous sulphate,
(U. S. P., pages 172 and 170).

Tests for identity:

Color—

Odor—

Taste—

Reaction to litmus—

+ $\text{K}_3\text{Fe}(\text{CN})_6$ T.S.—

+ BaCl_2 T.S. and HCl T.S.—

Tests for impurities:

Heavy metals—

Free acid—

TESTS FOR IDENTITY.

Granulated ferrous sulphate is a very pale bluish-green crystalline powder, without odor, and having a saline, styptic taste.

An aqueous solution of the salt (1 in 20) is acid to litmus.

An aqueous solution of the salt, even when highly diluted, gives with potassium ferricyanide T.S. a blue color or precipitate, and with barium chloride T.S. a white precipitate insoluble in hydrochloric acid.

TESTS FOR IMPURITIES.

Dissolve 1 Gm. of the salt in about 50 mls of distilled water containing 1 mil of diluted sulphuric acid, heat the solution to boiling, oxidize it with nitric acid and then mix it with a slight excess of ammonia water and filter. The filtrate is colorless, and, after acidulating with hydrochloric acid, it does not respond to the test for heavy metals (see general test No. 3, p. 118).

Agitate 1 Gm. of ferrous sulphate in small fragments during four or five minutes, with 10 mls of alcohol, and filter the mixture; the filtrate does not immediately redden moistened blue litmus paper (*free acid*).

SAMPLE NUMBER 22.**Examination of ammonium bromide for identity and purity.**
(U. S. P., p. 43.)

Tests should be carried out as described, and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Ammonium bromide occurs in colorless, transparent, prismatic crystals, or as a white, crystalline or granular powder; odorless, of a pungent, saline taste.

When heated, ammonium bromide volatilizes without fusing.

An aqueous solution of the salt (1 in 20) is neutral or not more than slightly acid to litmus.

An aqueous solution of ammonium bromide when gently heated with potassium hydroxide T.S. evolves ammonia.

Silver nitrate T.S. added to an aqueous solution of the salt (1 in 10) produces a yellowish-white precipitate, insoluble in nitric acid or in a moderate excess of ammonia water.

TESTS FOR IMPURITIES.

Add 1 mil of chloroform to 10 mls of an aqueous solution of the salt (1 in 20) and cautiously introduce chlorine water, which has been diluted with an equal volume of distilled water, drop by drop and with constant agitation. The liberated bromine dissolves in the chloroform and imparts to it a yellow or orange color, which is free from any violet tint (*iodide*).

Drop 1 mil of diluted sulphuric acid upon about 1 Gm. of the powdered salt; no yellow color appears at once (*bromate*).

A blue color is not produced at once on adding potassium ferrocyanide T.S. to 20 mls of an aqueous solution of the salt (1 in 250) (*iron*).

Add 1 mil of potassium sulphate T.S. to 10 mils of an aqueous solution of ammonium bromide (1 in 20), acidulated with acetic acid; no turbidity is produced immediately (*barium*); under similar conditions the addition of barium chloride T.S. produces no turbidity (*sulphate*).

An aqueous solution of the salt does not respond to the test for heavy metals (see general test No. 3, p. 118).

SAMPLE NUMBER 23.

Examination of diluted hydrochloric acid for identity and purity. (U. S. P., p. 14.)

The tests should be carried out as described, and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Diluted hydrochloric acid is a colorless, odorless liquid; it has a strongly acid taste and is strongly acid to litmus.

With silver nitrate T.S. it yields a white, curdy precipitate, insoluble in nitric acid, but readily soluble in ammonia water.

TESTS FOR IMPURITIES.

Add 1 mil of chloroform, to 10 mils of diluted hydrochloric acid and then cautiously introduce chlorine water; which has been diluted with an equal volume of distilled water, a drop at a time, with constant agitation; the chloroform remains free from any yellow, orange or violet color (*bromide or iodide*).

Add 1 mil of potassium iodide T.S. and 1 mil of chloroform to 10 mils of diluted hydrochloric acid and agitate the mixture; the chloroform remains free from any violet coloration (*free chlorine or bromine*).

Two mils of diluted hydrochloric acid, diluted with seven volumes of distilled water, does not respond to the test for heavy metals (see general test No. 3, p. 118).

Five mils of diluted hydrochloric acid diluted with 12 mils of distilled water (without the treatment with sulphuric and sulphurous acids) meets the requirements of the test for arsenic (see general test No. 1, p. 115).

Add 5 drops of barium chloride T.S. to a mixture of 3 mils of diluted hydrochloric acid and 5 mils of distilled water; neither turbidity nor precipitation appears within one hour (*sulphuric acid or sulphates*). At the end of this period, the further addition to the liquid of 2 drops of tenth-normal iodine V.S. produces neither turbidity nor decoloration of the iodine (*sulphurous acid*).

SAMPLE NUMBER 24.

Examination of bismuth subnitrate for identity and purity.

(U. S. P., pp. 82-83.)

The tests should be carried out as described and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Bismuth subnitrate is a white powder; odorless, almost tasteless.

Bismuth subnitrate is almost insoluble in water, insoluble in alcohol, and readily dissolved by hydrochloric or nitric acid.

Its solution in a slight excess of warm nitric or hydrochloric acid produces a white turbidity when added to 25 volumes of distilled water.

When heated to redness it evolves nitrous vapors, leaving a yellow residue, which is blackened by hydrogen sulphide T.S.

When brought in contact with moistened blue litmus paper, the salt shows a slightly acid reaction.

TESTS FOR IMPURITIES.

Boil about 1 Gm. of bismuth subnitrate with 20 mls of a mixture of equal parts of acetic acid and distilled water, cool the solution and filter. Free the filtrate from bismuth by the addition of hydrogen sulphide, boil the mixture and again filter. The latter filtrate leaves no appreciable residue on evaporation and gentle ignition (*alkalies or alkali earths*).

Boil about 0.1 Gm. of the salt with 5 mls of potassium hydroxide T.S.; no odor of ammonia is perceptible nor does the vapor turn moistened red litmus paper blue.

The residue resulting from the ignition of 2 Gm. of the salt does not respond to Bettendorf's test for arsenic (see general test No. 2, p. 117).

Add 3 Gm. of the salt to 3 mls of warm nitric acid; no effervescence occurs (*carbonate*), and no residue remains (*insoluble foreign salts*). Pour this solution into 100 mls of distilled water, a white precipitate is produced. Filter, evaporate the filtrate on a water-bath to 30 mls, again filter the liquid and divide the new filtrate into portions of 5 mls each.

Mix one portion with an equal volume of diluted sulphuric acid; it does not become cloudy (*lead*).

Precipitate another portion with a slight excess of ammonia water; the supernatant liquid does not exhibit a bluish tint (*copper*).

Another portion is not immediately affected by barium nitrate T.S. (*sulphate*). With hydrochloric acid no precipitate is formed

which is insoluble in a slight excess of the latter, but soluble in ammonia water (*silver*).

SAMPLE NUMBER 25.

Examination of alum for identity and purity. (U. S. P., pp. 39-40).

The tests should be carried out as described and a written record and report made as the work progresses, similar to that directed for sample No. 21.

TESTS FOR IDENTITY.

Ammonium alum and potassium alum both occur in large, colorless crystals, crystalline fragments, or as white powders; alum is odorless and has a sweetish and strongly astringent taste.

Ammonium alum.—Potassium hydroxide T.S. added to an aqueous solution of ammonium alum (1 in 20) at first causes a precipitate, which completely dissolves in an excess of the reagent, ammonia being evolved.

Potassium alum.—Potassium alum imparts a violet color to a non-luminous flame.

The addition of sodium bitartrate T.S. to a saturated solution of potassium alum produces, sometimes slowly, a white crystalline precipitate.

Potassium hydroxide T.S. added to an aqueous solution of potassium alum (1 in 20) at first causes a precipitate, which completely dissolves in an excess of the reagent, but no ammonia is evolved.

Both ammonium alum and potassium alum conform to the following tests:

An aqueous solution of alum (1 in 20) is acid to litmus.

An aqueous solution of alum (1 in 20) yields with ammonia water a white gelatinous precipitate almost insoluble in an excess of ammonia water.

With barium chloride T.S., an aqueous solution of alum (1 in 20) yields a white precipitate insoluble in hydrochloric acid.

TESTS FOR IMPURITIES.

An aqueous solution of alum does not respond to the test for heavy metals (see general test No. 3, p. 118). An aqueous solution of alum meets the requirements of the test for arsenic (see general test No. 1, p. 116).

Add 5 drops of potassium ferrocyanide T.S. to 20 mls of an aqueous solution of alum (1 in 150); no blue color is produced at once (*iron*).

GENERAL QUALITATIVE TESTS OF THE UNITED STATES PHARMACOPCEIA.

1. **Arsenic Test.**—*Test Apparatus.*—Prepare several generators, equipped with tubes, etc., as described below. Select as a generator a bottle of about 50 mils capacity, having a mouth about 2.5 cm. in diameter and provide a well-fitting rubber stopper, suitably perforated. In the perforation in this stopper insert a vertical exit tube about 13 cm. in total length and 1 cm. in diameter throughout the upper portion (for about 10 cm.) and constricted at its lower extremity to a tube of about 3 cm. in length and about 5 mm. in diameter. This latter tube should extend but slightly below the stopper. In the lower part of the wider exit tube insert a small pledget of dry glass wool and then a strip of the freshly prepared but dry lead acetate test-paper rolled into a coil, and above this a plug of the moist (not wet) lead acetate glass wool. In the upper extremity of this tube insert through a perforated cork stopper, a glass tube 12 cm. in length, having an internal diameter of about 3 mm. Place the mercuric bromide test-paper in this tube, bending or creasing the upper portion of the strip so that it will retain its position. The strip should extend within about 2 cm. of the perforated cork stopper and must not be introduced into the tube until ready to start the test. This tube should be thoroughly cleaned and dried each time it is used.

Standard Arsenic Test-solution.—Dissolve 0.1 Gm. of arsenic trioxide, which has been finely pulverized, dried in a desiccator, and accurately weighed, in about 5 mils of a 20 per cent. solution of sodium hydroxide (free from arsenic). Neutralize the solution with diluted sulphuric acid (free from arsenic), add 10 mils of the same acid and sufficient recently boiled distilled water to bring the volume of the solution to exactly 1000 mils at 25° C. Accurately measure 10 mils of this solution, transfer it to a 1000 mil flask, add 10 mils of diluted sulphuric acid (free from arsenic) and make up the volume with recently boiled distilled water to exactly 1000 mils at 25° C. Employ this solution, containing 0.001 mg. of arsenic trioxide in each 1 mil (at 25° C.), in preparing the standard stain. Keep this solution in a glass-stoppered bottle. It is advisable to make fresh solutions whenever new standard stains are to be prepared.

Preparation of the Chemical to be Tested.—Add 1 mil of a mixture of equal volumes of concentrated sulphuric acid and distilled water, to 5 mls of an aqueous solution of the chemical (1 in 25) or to a solution in 5 mls of distilled water of the residue remaining after any special treatment that may be directed. This acidulation is not necessary in the case of inorganic acids. Now, unless specially directed, add 10 mls of a saturated aqueous solution of sulphurous acid. Evaporate this liquid in a small beaker, on a water-bath, until it is free from sulphurous acid and has been reduced to about 2 mls in volume. Dilute this evaporated liquid to about 5 mls with distilled water.

THE TEST FOR ARSENIC.—Preparation of the Standard Stain.—Introduce into the generator from 8 to 10 Gm. of zinc followed by 25 mls of dilute sulphuric acid, prepared by mixing 1 part of concentrated sulphuric acid, free from arsenic, with 4 parts of distilled water, and 5 drops of acid stannous chloride T.S. Add at once 2 mls (accurately measured at 25° C.) of the standard arsenic T.S. and immediately insert the stopper containing the exit tube, into which have been placed the glass-wool pledget, the dry lead acetate test-paper, the moist lead acetate glass wool, and the mercuric bromide test-paper, as described under the *Test Apparatus*. Should the evolution of the gas be violent at first, check the reaction by immersing the bottle in cold water. Should the reaction subside, increase it by placing the bottle in warm water. If the reaction be too violent, the stain will spread and not form a distinctive band, thus making the comparisons of color intensity difficult. After the test has continued for forty-five minutes, remove the mercuric bromide test-paper and place it in a clean, dry tube for comparison. This stain represents 0.002 mg. of arsenic trioxide in addition to any stain produced by the reagents. The stain from the reagents should scarcely be perceptible when determined by a blank experiment. Since light, heat and moisture cause the stain to fade rapidly, comparison should be made as soon as possible. The stained test-papers may be preserved by either dipping in hot, melted paraffin or keeping them over phosphoric anhydride protected from light.

Testing the Chemical.—Introduce into another generator from 8 to 10 Gm. of the zinc, followed by 25 mls of the dilute sulphuric acid (1 to 4) and 5 drops of acid stannous chloride T.S. Insert the stopper containing the exit tube charged with the test-papers and glass wool, as just described. Then add 5 mls of the solution to be tested, previously treated as directed, under *Preparation of the Chemical*, and immediately insert the stopper charged with the test-papers and glass wool. When the evolution of hydrogen has pro-

ceeded actively for forty-five minutes, remove the mercuric bromide test-paper and carefully compare it with the standard stain prepared as described above. The stain produced by the chemicals tested does not exceed in length or intensity of color that prepared as the standard, indicating not more than 1 part of arsenic in 100,000 parts of the substance tested.

Interfering Chemicals.—*Antimony*, if present in the substance tested, will produce a gray stain. *Sulphites, sulphides, thiosulphates*, and other compounds which liberate hydrogen sulphide or sulphurous acid when treated with sulphuric acid, must be oxidized by means of nitric acid and then reduced by means of sulphurous acid as directed under *Preparation of the chemical to be tested*, before introducing them into the apparatus. *Sulphur compounds* as well as *hydrogen phosphide* give a bright yellow band on the test paper. If sulphur compounds are present, a simultaneous darkening of the lead acetate test paper and glass wool will occur. If such is the case, the operation as directed under *Preparation of the chemical to be tested* must be repeated upon a fresh portion of the solution being tested, using greater care in effecting the complete removal of the sulphurous acid. In testing hypophosphites special care should be observed to completely oxidize the solution being tested as directed, otherwise a yellow stain may be produced through the evolution of hydrogen phosphide, which might be confused with the orange-yellow color produced by arsenic. *Compounds containing antimony* should be tested for *arsenic* by Bettendorf's Test (see Test No. 2).

The test apparatus should be thoroughly cleaned and dried immediately before and after use.

2 Arsenic test, Bettendorf's.—This test is employed only in testing salts of *bismuth* and compounds containing *antimony* for the presence of arsenic.

To a solution of the prescribed quantity of the substance to be tested in 5 mls of concentrated hydrochloric acid contained in a clean test tube, add 10 mls of saturated stannous chloride T.S. which has been freshly prepared, and set it aside for thirty minutes. If arsenic is present in non-permissible amounts, a brownish tint or precipitate will be seen when the tube is placed over a white surface and the solution viewed from above, comparison being made with a mixture of 5 mls of concentrated hydrochloric acid and 10 mls of concentrated stannous chloride T.S., prepared as directed above.

NOTE: It is absolutely necessary that for this test the solution of stannous chloride be freshly prepared, and that nitrates, sulphates, sulphites, sulphides and compounds of mercury, gold, and selenium be absent from the reagents, and from the chemicals being tested.

3 Test for heavy metals.—Chemicals.—This test is to be used to detect the presence of undesirable metallic impurities in official chemical substances or their solutions; these must not respond affirmatively within the stated time. Acidulate 10 mls of a solution of the substance in distilled water (1 in 50), contained in a test tube of about 40 mls capacity and of about 2.5 cm. diameter, with 1 ml of diluted hydrochloric acid (unless otherwise directed), warm it to about 50° C., add an equal volume of freshly prepared hydrogen sulphide T.S., stopper, and allow the mixture to stand at 35° C. for half an hour. At the end of this time the mixture should still possess the odor of hydrogen sulphide; if not, it should be thoroughly saturated with the gas and again set aside for half an hour. The color produced, if any, is not greater than that observed by a blank test made in the same manner and with the same quantities of the reagents (omitting the solution to be tested); the solutions being viewed crosswise by reflected light while held against a white surface. A slight turbidity due to separation of sulphur from the hydrogen sulphide may occur.

DESTRUCTION OF ORGANIC MATTER.

If organic matter is present in a sample, it is necessary to destroy it before proceeding with a qualitative analysis for inorganic bases and acids. There are several methods for accomplishing this, of which the most generally useful are the following:

A. Mix the sample with an excess of concentrated HCl, add some crystals of KClO_3 and boil under a hood until the mixture is decolorized or a portion does not carbonize upon drying and igniting. Add additional HCl and KClO_3 as necessary, avoiding a large excess. Then evaporate the liquid to dryness on a water bath and examine the residue by the method for the qualitative analysis of a solid.

This method destroys the organic matter, or changes it so that it does not interfere with the analysis. The metals present are oxidized to their highest valences, and ammonium salts are volatilized. Allowance must also be made for the potassium and chlorine compounds added.

B. Mix the sample with about 4 times its weight of concentrated H_2SO_4 and twice its weight of concentrated HNO_3 , digest at the ordinary temperature for several hours, and then heat in a hood until the white fumes of H_2SO_4 are given off. If the mixture still contains black carbonized organic matter, add more HNO_3 and repeat the heating until white fumes are given off, repeating this treatment with additional HNO_3 and heating several times if necessary. When the mixture ceases to show the presence of carbon, cautiously add about an equal volume of water and concentrate until the white fumes of H_2SO_4 are given off, then cautiously add four or five times the volume of water and filter, if necessary. Examine the filtrate by the method for the qualitative analysis of a liquid. Any residue, on the filter paper, may contain silica or the sulphates of lead, barium, strontium and calcium. Examine it by the method for the qualitative analysis of a solid, beginning with paragraph F on page 108.

This is the most dependable method for the destruction of organic matter. Allowance must be made, however, for the H_2SO_4 and HNO_3 used, and it is often necessary to use both of these methods, or others, in making a careful qualitative analysis of a substance containing organic matter.

REAGENTS AND TEST SOLUTIONS.

On the following pages will be found directions for preparing the test solutions called for in this book. Most of these are of the same strength as is specified in the United States Pharmacopœia, but for performing the Pharmacopœial tests on official chemicals, the reagents should be made according to the directions of the Pharmacopœia IX, pages 521-583.

In preparing reagents and test solutions, only pure chemicals and distilled water should be used.

Acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$.—Mix official acetic acid, containing about 36 per cent. of absolute $\text{HC}_2\text{H}_3\text{O}_2$ with twice its volume of distilled water.

Ammonium hydroxide test solution, NH_4OH or $\text{NH}_3 + \text{H}_2\text{O}$.—Use the official ammonia water, containing 10 per cent. of NH_3 .

Ammonium carbonate test solution, $(\text{NH}_4)_2\text{CO}_3$.—Dissolve 20 Gm. of official ammonium carbonate in a mixture of 20 mls of ammonia water and 65 mls of distilled water, and add sufficient distilled water to measure 100 mls.

Ammonium chloride test solution, NH_4Cl .—Dissolve 10 Gm. of ammonium chloride in sufficient distilled water to make 100 mls.

Ammonium molybdate test solution, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} + 4\text{H}_2\text{O}$.—Mix 6.5 Gm. of finely powdered molybdic acid with 14 mls of distilled water and 14.5 mls of stronger ammonia water to effect solution. Cool and slowly add the solution, in small portions with agitation, to a well cooled mixture of 32 mls of nitric acid and 40 mls of distilled water. Allow the solution to stand for twenty-four hours and then filter through asbestos.

Preserve the test solution in the dark, and, if a sediment should form in it after some days, carefully decant the clear solution. This solution should be tested at frequent intervals. Add 2 mls of sodium phosphate T.S. to 5 mls of the reagent; an abundant yellow precipitate forms either at once or upon slight warming. If only a slight precipitation or yellow opalescence results, the reagent must be rejected. When employed as a reagent, ammonium molybdate T.S. is always added in large excess to the solution being tested, the latter having previously been strongly acidified with nitric acid.

Ammonium oxalate test solution $(\text{NH}_4)_2\text{C}_2\text{O}_4 + \text{H}_2\text{O}$.—Dissolve 4 Gm. of ammonium oxalate in sufficient distilled water to measure 100 mls.

Ammonium polysulphide test solution, Yellow Ammonium Sulphide, $(\text{NH}_4)_2\text{S}_x$.—Saturate three parts of ammonia water with hydrogen sulphide, forming a solution of ammonium hydrogen sulphide, NH_4HS . Add to the solution two additional parts of ammonia water, forming a solution of normal or colorless ammonium sulphide, $(\text{NH}_4)_2\text{S}$. Finally add a small quantity of precipitated sulphur and preserve the solution in small, amber, glass-stoppered bottles.

Barium chloride test solution, $\text{BaCl}_2 + 2\text{H}_2\text{O}$.—Dissolve 10 Gm. of barium chloride in sufficient distilled water to make 100 mls.

Barium hydroxide test solution, $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$.—A freshly made saturated solution of barium hydroxide in distilled water.

Barium nitrate test solution, $\text{Ba}(\text{NO}_3)_2$.—Dissolve 5 Gm. of barium nitrate in sufficient distilled water to make 100 mls.

Bromine test solution (bromine water), Br.—A saturated solution of bromine, prepared by adding 3 mls of bromine to 100 mls of cold water. The solution should be stored in a glass-stoppered bottle in a cool place, protected from light and should be shaken well, allowing the excess of bromine to settle before using.

Calcium chloride test solution, $\text{CaCl}_2 + 2\text{H}_2\text{O}$.—Dissolve 10 Gm. of calcium chloride in sufficient distilled water to measure 100 mls.

Calcium hydroxide test solution (lime water), $\text{Ca}(\text{OH})_2$.—A nearly saturated aqueous solution of calcium hydroxide.

Calcium sulphate test solution, $\text{CaSO}_4 + 2\text{H}_2\text{O}$.—Mix 5 mls of diluted sulphuric acid with 200 mls of distilled water, add 1 Gm. of calcium carbonate and shake: when the acid is completely neutralized filter the mixture.

Chlorine test solution (chlorine water), Cl.—A saturated aqueous solution of chlorine, prepared by generating the gas with manganese and hydrochloric acid, washing and conducting it into cold water until saturated. The solution should be preserved in small amber glass-stoppered bottles in a cool, dark place and it should have a strong odor of chlorine when used.

Cobaltous chloride test solution, $\text{Co}_2\text{Cl} + 6\text{H}_2\text{O}$.—Dissolve 2 Gm. of cobaltous chloride with the aid of 1 mil of hydrochloric acid in sufficient distilled water to measure 100 mls.

Copper sulphate test solution, $\text{CuSO}_4 + 5\text{H}_2\text{O}$.—Dissolve 10 Gm. of copper sulphate in sufficient distilled water to make 100 mls.

Ferric chloride test solution, $\text{FeCl}_3 + 6\text{H}_2\text{O}$.—Dissolve 10 Gm. of ferric chloride in sufficient water to make 100 mls.

Ferrous sulphate test solution, $\text{FeSO}_4 + 7\text{H}_2\text{O}$.—Dissolve 10 Gm. of clear crystals of ferrous sulphate in sufficient distilled water to make 100 mls. This solution is to be freshly prepared immediately before use.

Hydrochloric acid test solution, HCl.—Use the official diluted hydrochloric acid of 10 per cent. strength.

Hydrogen sulphide test solution, H_2S .—A saturated, aqueous solution of hydrogen sulphide. To prepare about 1000 mls of the solution, treat 20 Gm. of ferrous sulphide in a suitable apparatus with a mixture of 20 mls of sulphuric acid and 250 mls of distilled water, pass the gas through a drying tube filled with granulated calcium chloride, then from this through a tube of about 8 millimeters diameter and 40 centimeters in length, which contains about 5 Gm. of coarsely pulverized iodine mixed with glass wool, and finally through a wash bottle which contains a small quantity of potassium iodide T.S. The gas thus purified is conducted nearly to the bottom of a bottle of the capacity of about 1500 mls containing 1000 mls of cold distilled water. Shake the bottle occasionally to facilitate the solution of the gas. When the gas is no longer absorbed, transfer the solution to small, dark, amber-colored bottles, fill nearly to the top; pass a stream of purified hydrogen sulphide for a few minutes through each, and then at once stopper them tightly, and preserve them afterwards in a cool and dark place. Do not use this solution unless it retains a strong odor of hydrogen sulphide, and, when added to an equal volume of ferric chloride T.S., produces at once a copious precipitate of sulphur.

Iodine test solution, I.—Dissolve 1 Gm. of iodine and 3 Gm. of potassium iodide in 50 mls of distilled water.

Lead acetate glass wool, $Pb(C_2H_3O_2)_2 + 3H_2O$.—Immerse glass wool in a mixture of equal parts of lead acetate T.S. and water and remove the excess of liquid by pressing it between filter paper. It should be prepared immediately before it is to be used.

Lead acetate test paper, $Pb(C_2H_3O_2)_2 + 3H_2O$.—Immerse strips of heavy white filter paper, 6 cm. in width and 8 cm. in length, in a mixture of equal parts of lead acetate T.S. and distilled water, drain off the excess of liquid and dry the paper in an oven at $100^\circ C.$, avoiding contact with metal.

Lead acetate test solution, $Pb(C_2H_3O_2)_2 + 3H_2O$.—Dissolve 10 Gm. of clear transparent crystals of lead acetate, free from adhering lead carbonate, in sufficient distilled water to make 100 mls. Preserve the solution in well-stoppered bottles.

Litmus paper and test solution.—Exhaust powdered litmus with three separate and successive portions (each equal to about 4 times its weight) of boiling alcohol (which removes the undesirable color erythrolitmin), each extraction lasting for about one hour. After draining off the alcohol, digest the residue with about an equal weight of cold water and filter. (This blue solution, which contains some alkali, after being acidulated, may be used to make red litmus paper.) Finally, extract the residue with about 5 times its weight of boiling distilled water, and, after thoroughly cooling, filter. The

addition of 1 drop of hundredth-normal acid or alkali V.S. to 50 mls of distilled water containing 5 drops of the indicator produces a distinct change in color. Preserve the filtrate, as a test solution, in wide-mouthed bottles stoppered with loose plugs of purified cotton so as to exclude dust but admit air. The latter must be free from acid or ammoniacal vapors. The blue color of litmus test solution is changed by acids to red, and this red color by the addition of alkalies is restored to blue.

Litmus Paper, Blue.—Impregnate with the test solution just described strips of white filter paper, and dry them by suspending them on lines of clean twine, in an atmosphere free from acid or ammoniacal vapors. This paper must quickly respond to a two hundred and fiftieth-normal acid V.S.

Litmus Paper, Red.—Prepare this with the same kind of paper and in the manner described under *Litmus Paper, Blue*, having added to the test solution used to impregnate the paper just sufficient of a highly diluted solution of hydrochloric acid to impart to it a faint red tint. Neither blue nor red litmus paper should have an intense color. Preserve the test paper in bottles, so as to exclude dust and acid or ammoniacal vapors. This paper must quickly respond to a two hundred and fiftieth-normal alkali V.S.

Magnesia mixture, $\text{MgCl}_2 + 6\text{H}_2\text{O}$, NH_4Cl , NH_4OH .—Dissolve 5.5 Gm. of magnesium chloride and 7 Gm. of ammonium chloride in 65 mls of distilled water, add 35 mls of ammonia water, set the mixture aside for a few days in a well-stoppered vessel, and filter. If not perfectly clear, filter the solution before using.

Magnesium sulphate test solution, $\text{MgSO}_4 + 7\text{H}_2\text{O}$.—Dissolve 10 Gm. of magnesium sulphate in sufficient distilled water to measure 100 mls.

Manganese sulphate test solution, $\text{MnSO}_4 + 4\text{H}_2\text{O}$.—Dissolve 10 Gm. of manganese sulphate in 50 mls of distilled water and add sufficient diluted sulphuric acid to make 100 mls.

Mercuric bromide test paper, HgBr_2 .—Cut stiff, heavy quantitative filter paper into strips 3 mm. in width and about 12 cm. in length. Immerse these strips for five minutes in alcoholic mercuric bromide T.S. Remove the excess of solution by pressing the strips between filter paper and then dry them quickly on glass in an oven heated to 100°C . Place the strips at once in a wide mouthed bottle and stopper it securely.

Mercuric bromide test solution, alcoholic, HgBr_2 .—Dissolve 5 Gm. of mercuric bromide in 100 mls of alcohol, employing a gentle heat to facilitate solution. Keep it in glass stoppered bottles protected from the light.

Mercuric chloride test solution, HgCl_2 .—Dissolve 5 Gm. of mercuric chloride in sufficient distilled water to measure 100 mls.

Mercuric potassium iodide test solution (*Mayer's reagent*), $\text{HgI}_2 + 2\text{KI}$.—Dissolve 1.358 Gm. of mercuric chloride in 60 mls of distilled water, and 5 Gm. of potassium iodide in 10 mls of distilled water. Mix the two solutions, and then add sufficient distilled water to measure 100 mls.

Mercuric potassium iodide test solution, alkaline (*Nessler's Reagent*).—Dissolve 10 Gm. of potassium iodide in 10 mls of distilled water, and add gradually in portions a saturated aqueous solution of corrosive mercuric chloride with constant agitation, until a slight red precipitate remains undissolved; to this mixture add 30 Gm. of potassium hydroxide and, when solution has taken place, 1 ml more of the saturated aqueous solution of mercuric chloride. Dilute this solution with distilled water until it measures 200 mls. Allow the precipitate to subside, and draw off the clear fluid. Two mls of this reagent, when added to 50 mls of distilled water containing 0.05 mg. of ammonia, produces at once a yellowish brown coloration.

Mercurous nitrate test solution, HgNO_3 .—Mix 10 Gm. of pure mercury with 5 mls of nitric acid and 5 mls of distilled water, in a porcelain evaporating dish and set it aside for twenty-four hours in a cool, dark room. Separate and drain the crystals of mercurous nitrate, and dissolve them in 100 mls of distilled water. Preserve the solution in a dark amber-colored bottle in which a small quantity of mercury has been placed.

Nitric acid test solution, HNO_3 .—Add 36 mls of concentrated nitric acid to 290 mls of distilled water and mix. This solution contains approximately 10 per cent. of absolute HNO_3 .

Oxalic acid test solution, $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$.—Dissolve 5 Gm. of oxalic acid in sufficient distilled water to measure 100 mls.

Phenolphthalein test solution.—Dissolve 1 Gm. of phenolphthalein in 100 mls of alcohol. It gives a red color with alkali hydroxides or carbonates, and acids render the solution colorless. It is not suitable as an indicator for ammonia nor in the presence of large quantities of ammonium salts. Phenolphthalein paper is prepared by impregnating white, unsized paper with the test solution and drying it.

Potassium carbonate test solution, K_2CO_3 .—Dissolve 10 Gm. of potassium carbonate in sufficient distilled water to measure 100 mls.

Potassium chloride test solution, KCl .—Dissolve 10 Gm. of potassium chloride in sufficient distilled water to measure 100 mls.

Potassium chromate test solution, K_2CrO_4 .—Dissolve 10 Gm. of potassium chromate in sufficient distilled water to measure 100 mls.

Potassium cyanide test solution, KCN .—Dissolve 5 Gm. of potassium cyanide in sufficient distilled water to measure 100 mls.

Potassium dichromate test solution, $\text{K}_2\text{Cr}_2\text{O}_7$.—Dissolve 10 Gm. of potassium dichromate in sufficient distilled water to measure 100 mls.

Potassium ferricyanide test solution, $K_3Fe(CN)_6$.—Dissolve 1 part of potassium ferricyanide in about 10 parts of distilled water. This solution should be freshly made when required, as it undergoes decomposition with formation of ferrocyanide on standing. A freshly prepared aqueous solution mixed with ferric chloride T.S. which has been well diluted with distilled water shows a brown tint, free from turbidity or a shade of green.

Potassium ferrocyanide test solution, $K_4Fe(CN)_6 + 3H_2O$.—Dissolve 10 Gm. of potassium ferrocyanide in sufficient distilled water to measure 100 mils.

Potassium hydroxide test solution, KOH.—Use the official solution of potassium hydroxide containing approximately 5 per cent. of KOH.

Potassium iodide test solution, KI.—Dissolve 20 Gm. of potassium iodide in sufficient distilled water to measure 100 mils, and preserve the solution in dark, amber-colored, well-stoppered bottles. The solution should be frequently renewed.

Potassium sulphate test solution, K_2SO_4 .—Dissolve 1 Gm. of potassium sulphate in sufficient distilled water to measure 100 mils.

Potassium sulphocyanate test solution, KCNS.—Dissolve 1 Gm. of potassium sulphocyanate in sufficient distilled water to make 100 mils.

Silver nitrate test solution, $AgNO_3$.—Dissolve 2 Gm. of silver nitrate in sufficient distilled water to measure 100 mils.

Sodium acetate test solution, $NaC_2H_3O_2 + 3H_2O$.—Dissolve 10 Gm. of sodium acetate in sufficient distilled water to make 100 mils.

Sodium carbonate test solution, $Na_2CO_3 + H_2O$.—Dissolve 10 Gm. of monohydrated sodium carbonate in sufficient distilled water to measure 100 mils.

Sodium cobaltic nitrite test solution, $(NaNO_2)_6 Co_2(NO_2)_6 + H_2O$.—Dissolve 4 Gm. of cobaltous chloride and 10 Gm. of sodium nitrite in about 50 mils of distilled water, add 2 mils of acetic acid and dilute with sufficient distilled water to measure 100 mils. A few drops of acetic acid should be added to the solution from time to time. The reagent must not be kept longer than three months. Should any precipitate form on standing, filter.

Sodium cyanide test solution, NaCN.—Dissolve 1 Gm. of sodium cyanide in sufficient distilled water to measure 10 mils. The solution must be freshly prepared when required.

Sodium hydroxide test solution, NaOH.—Dissolve 10 Gm. of sodium hydroxide in sufficient distilled water to measure 100 mils.

Sodium hypobromite test solution, NaBrO.—To a solution of 40 Gm. of sodium hydroxide in about 150 mils of distilled water add 10 mils of bromine and, after solution has taken place, add sufficient distilled water to measure 200 mils. The solution must be freshly prepared when required for use.

Sodium nitroprusside test solution, $\text{Na}_2\text{FeNO}(\text{CN})_5 + 2\text{H}_2\text{O}$.—Dissolve 1 part of sodium nitroprusside in 19 parts of distilled water immediately before using.

Sodium phosphate test solution, $\text{NaHPO}_4 + 12\text{H}_2\text{O}$.—Dissolve 10 Gm. of sodium phosphate, in clear crystals, in sufficient distilled water to measure 100 mls.

Sodium tartrate test solution, $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 + 2\text{H}_2\text{O}$.—Dissolve 10 Gm. of sodium tartrate in sufficient distilled water to measure 100 mls.

Sodium thiosulphate test solution, $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$.—Dissolve 2.5 Gm. of sodium thiosulphate in sufficient distilled water to measure 100 mls.

Stannous chloride test solution, $\text{SnCl}_2 + 2\text{H}_2\text{O}$.—Dissolve 10 Gm. of stannous chloride crystals in 100 mls of distilled water to which a small amount of hydrochloric acid has been added, and preserve the solution in glass-stoppered bottles in which a fragment of tin has been placed. The solution must be renewed at frequent intervals.

Stannous chloride test solution, saturated, $\text{SnCl}_2 + 2\text{H}_2\text{O}$.—(For Bettendorf's test for arsenic.) A saturated solution of stannous chloride crystals in concentrated hydrochloric acid. After filtering the solution through asbestos it has not more than a pale yellow color.

Starch test solution.—Triturate 1 Gm. of cornstarch with 10 mls of cold distilled water, add boiling distilled water with constant stirring to make about 200 mls, then boil the mixture for a few minutes until a thin, translucent fluid is obtained. This solution must be freshly prepared when required.

Sulphuric acid test solution, H_2SO_4 .—Use the official diluted sulphuric acid, containing about 10 per cent. of absolute H_2SO_4 .

Tartaric acid test solution, $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$.—Dissolve 1 part of tartaric acid in 3 parts of distilled water. This solution must be frequently renewed.

Turmeric tincture.—Digest any convenient quantity of ground turmeric root repeatedly with small quantities of distilled water and discard the liquids. Then digest the dried residue for several days with six times its weight of alcohol, and filter.

Turmeric paper.—Impregnate white, unsized paper with the tincture, and dry it. The tincture, as well as the paper, turns brown with alkalies, and the original yellow color is restored by acids, with the exception of boric acid, which, especially in the presence of hydrochloric acid, turns the color to reddish-brown, which is changed to bluish-black by ammonia.

Zinc sulphate test solution, ZnSO_4 .—Dissolve 10 Gm. of zinc sulphate in sufficient distilled water to measure 100 mls.

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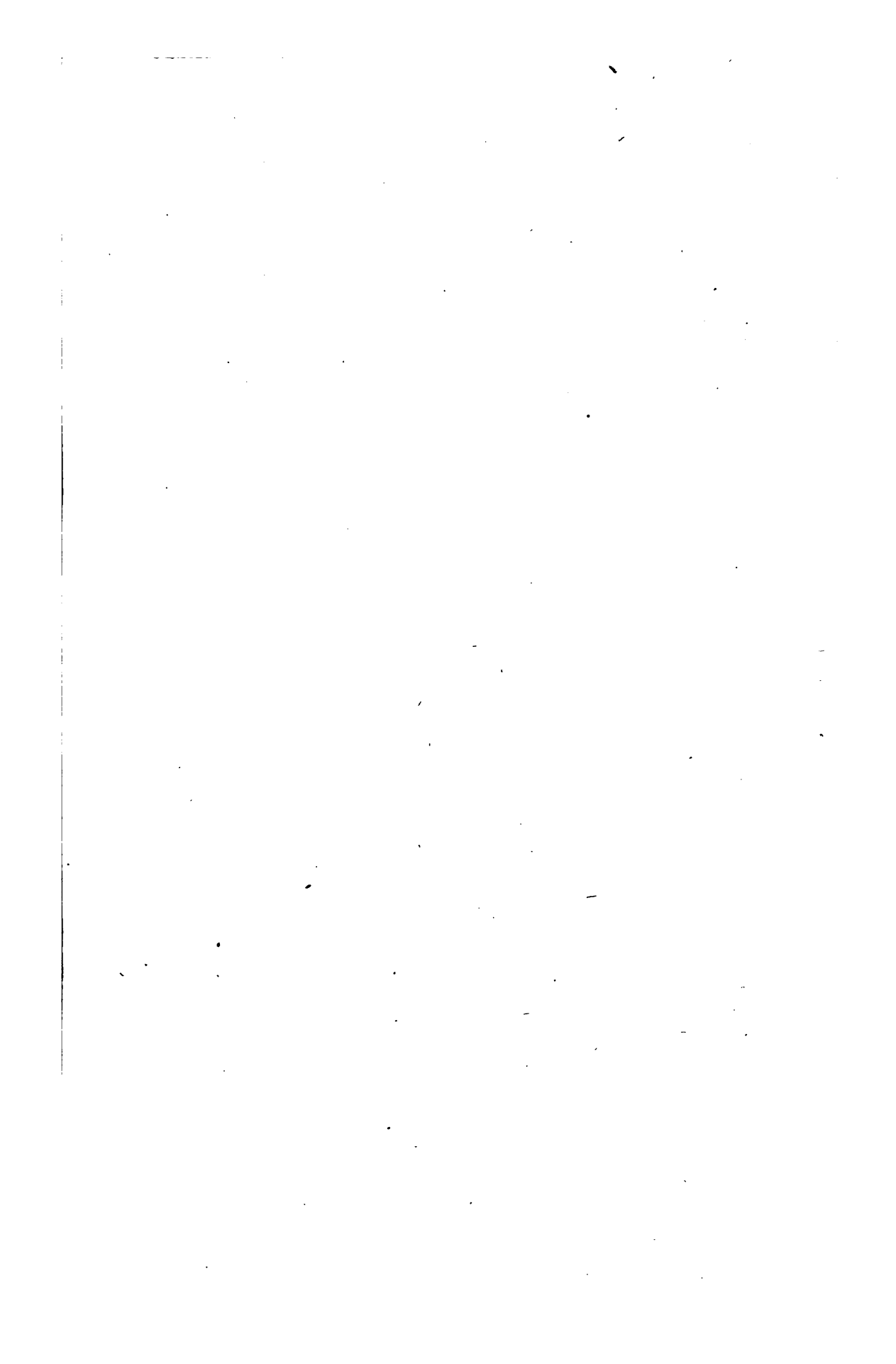
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